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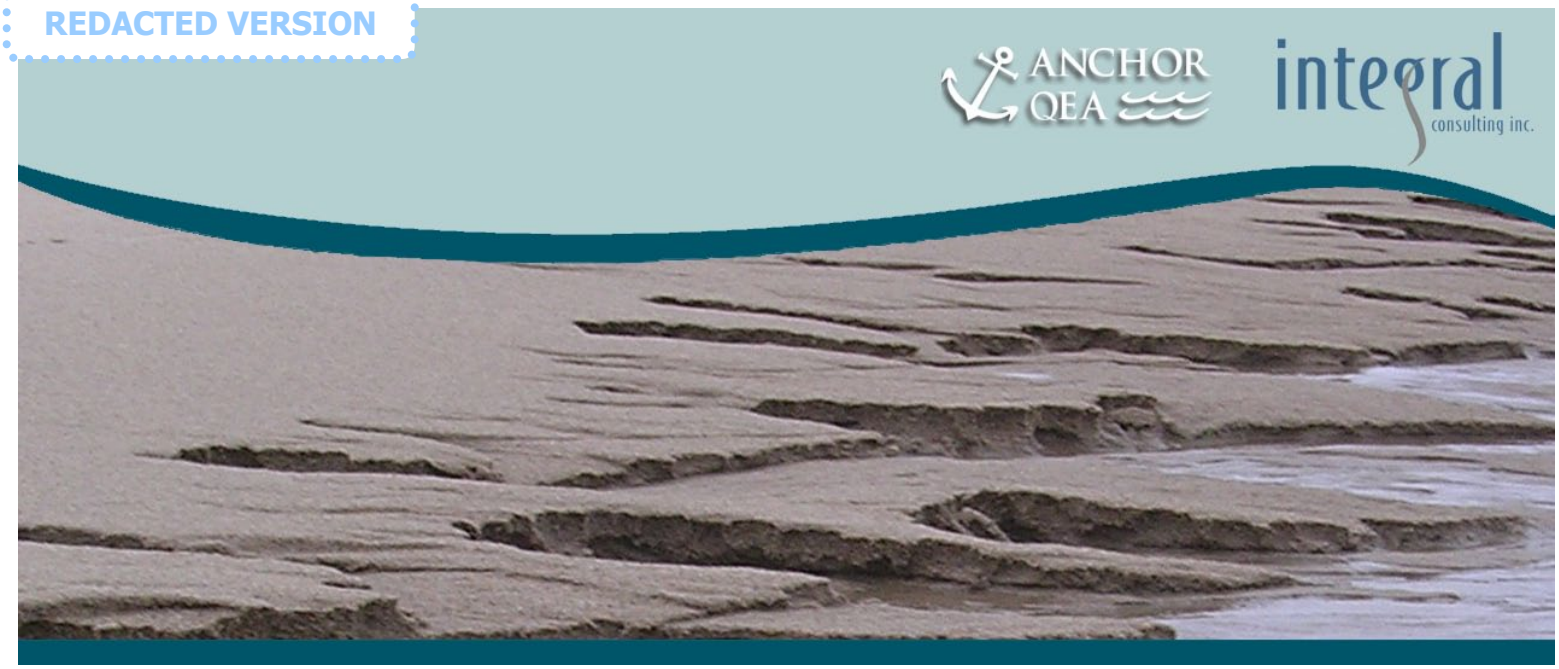

DRAFT SAMPLING AND ANALYSIS PLAN: SOIL STUDY
SAN JACINTO RIVER WASTE PITS SUPERFUND SITE

Prepared for

McGinnes Industrial Maintenance Corporation
International Paper Company
U.S. Environmental Protection Agency, Region 6

Prepared by

Integral Consulting Inc.
411 First Avenue South, Suite 550
Seattle, Washington 98104

December 2010

9546106

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Title and Approval Sheet

Quality Assurance Project Plan Approvals

USEPA Remedial Project Manager:	Stephen Tzhone	_____	Date: _____
USEPA Quality Assurance (QA) Reviewer:	Walter Helmick	_____	Date: _____
Respondents' Project Coordinator and Anchor QEA Project Manager:	David Keith	_____	Date: _____
McGinnes Industrial Maintenance Corp. Project Manager:	Andrew Shafer	_____	Date: _____
International Paper Co. Project Manager:	Philip Slowiak	_____	Date: _____
Integral Project Manager:	Jennifer Sampson	_____	Date: _____
Laboratory QA Coordinator:	Craig Hutchings	_____	Date: _____
Chemical Laboratory Project Manager (CAS Kelso):	Greg Salata	_____	Date: _____
Chemical Laboratory QA Manager (CAS Kelso):	Julie Gish	_____	Date: _____

Chemical Laboratory Project Manager (CAS Houston, HRMS Analyses):	Darren Biles	_____	Date: _____
Chemical Laboratory QA Manager (CAS Houston, HRMS Analyses):	Andrew Biddle	_____	Date: _____

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LIST OF ACRONYMS AND ABBREVIATIONS

Anchor QEA	Anchor QEA, LLC
ASTM	American Society for Testing and Materials
COC	chain-of-custody
COI	chemical of interest
COPC	chemical of potential concern
CLP	Contract Laboratory Program
CSM	conceptual site model
CT	central tendency
DQO	Data Quality Objective
dw	dry weight
EDD	electronic data deliverable
EDL	estimated detection limit
FS	Feasibility Study
FSP	Field Sampling Plan
GPS	global positioning system
HAZWOPER	Hazardous Waste Operations and Emergency Response
HRGC/HRMS	high-resolution gas chromatography with high-resolution mass spectrometry
HASP	Health and Safety Plan
HRMS	high-resolution mass spectrometry
I-10	Interstate Highway 10
Integral	Integral Consulting Inc.
IPC	International Paper Company
LiDAR	light detection and ranging
MIMC	McGinnes Industrial Maintenance Corporation
MDL	method detection limit
MRL	method reporting limit
NPL	National Priorities List
PARCC	precision, accuracy or bias, representativeness, completeness, comparability
PCA	principal components analysis

PCB	polychlorinated biphenyl
PCL	Protective Concentration Level
PPL	priority pollutant list
PRG	Preliminary Remediation Goal
PSCR	Preliminary Site Characterization Report
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RAWP	Removal Action Work Plan
RI	Remedial Investigation
RME	reasonable maximum exposure
ROW	right-of-way
RPD	relative percent difference
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
Site	San Jacinto River Waste Pits Superfund Site
SJRWP	San Jacinto River Waste Pits
SOP	standard operating procedure
SRM	Standard Reference Material
SVOC	semivolatile organic compound
TAL	target analyte list
TCEQ	Texas Commission on Environmental Quality
TCRA	Time Critical Removal Action
TEF	toxicity equivalence factor
TEQ_{DF}	toxicity equivalence concentrations calculated with dioxins and furans only
TMDL	Total Maximum Daily Load
TOC	total organic carbon
TSDH	Texas State Department of Health
TxDOT	Texas Department of Transportation
UAO	Unilateral Administrative Order
UCL	upper confidence limit
Unmixing	unmixing analysis

UPL	upper prediction limit
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
UTL	upper tolerance limit

1 PROJECT MANAGEMENT

1.1 Distribution List

Title	Name
USEPA Remedial Project Manager	Stephen Tzhone
USEPA QA Reviewer	Walter Helmick
Respondents' Project Coordinator and Anchor QEA Project Manager	David Keith
McGinnes Industrial Maintenance Corp. Project Manager	Andrew Shafer
International Paper Co. Project Manager	Philip Slowiak
Integral Project Manager	Jennifer Sampson
Field Lead	Bill Lawrence
Laboratory QA Coordinator	Craig Hutchings
Database Administrator	Dreas Nielsen
Chemical Testing Laboratory Project Manager (CAS Kelso)	Greg Salata
Chemical Testing Laboratory QA Manager (CAS Kelso)	Julie Gish
Chemical Testing Laboratory Project Manager (CAS Houston)	Darren Biles
Chemical Testing Laboratory QA Manager (CAS Houston)	Andrew Biddle

1.2 Introduction and Task Organization

This Sampling and Analysis Plan (SAP) has been prepared on behalf of International Paper Company (IPC) and McGinnes Industrial Maintenance Corporation (MIMC), pursuant to the requirements of Unilateral Administrative Order (UAO), Docket No. 06-03-10, which was issued by the U.S. Environmental Protection Agency (USEPA) to IPC and MIMC on November 20, 2009 (USEPA 2009c). The 2009 UAO directs IPC and MIMC to conduct a Remedial Investigation and Feasibility Study (RI/FS) for the San Jacinto River Waste Pits (SJRWP) Superfund Site in Harris County, Texas (the Site).

This document presents the Soil SAP consisting of a Quality Assurance Project Plan (QAPP) and the Field Sampling Plan (FSP), which is included as Appendix A. The QAPP was prepared consistent with USEPA guidance and requirements for QAPPs (USEPA 2001a, 2002b), as required by the 2009 UAO. Together, these components describe the soil study, which will be used to inform the RI/FS required by the 2009 UAO.

This section reviews the organizational structure for activities associated with the soil study, including project management and oversight, fieldwork, sample analysis, and data management. The organizational structure for this project is illustrated in Figure 1. Contact information for key personnel is provided in Section 1.3.

1.3 Project Organization

IPC and MIMC have retained Integral Consulting Inc. (Integral) and Anchor QEA, LLC (Anchor QEA) to perform the activities associated with execution of the Soil SAP. Figure 1 illustrates the organization of personnel on the project. The primary contacts for USEPA, MIMC, and IPC are provided in the following table. A description of the project organization and contacts pertaining to this QAPP are provided after the table.

USEPA and Respondent Project Managers

Title	Name	Contact Information
USEPA Remedial Project Manager	Stephen Tzhone	U.S. Environmental Protection Agency, Region 6 1445 Ross Avenue Dallas, TX 75202-2773 (214) 665-8409 tzhone.stephen@epa.gov
McGinnes Industrial Maintenance Corporation Project Manager	Andrew Shafer	9590 Clay Road Houston, TX 77080 (713) 772-9100, ext. 109 DShafer@wm.com
International Paper Company Project Manager	Philip Slowiak	6400 Poplar Avenue Memphis, TN 38197-0001 (901) 419-3845 philip.slowiak@ipaper.com

To execute this study, Integral and Anchor QEA will conduct the fieldwork, database administration, coordination with the laboratories, and data analysis. The names and quality assurance (QA) responsibilities of key project personnel who will be involved in sampling and analysis activities are provided below. For addenda to this SAP, Figure 1 and these two tables will be revised and presented as appropriate for the tasks described in each addendum.

Project Personnel Quality Assurance Responsibilities

Title	Responsibility	Name	Contact Information
Project Coordinator and Anchor QEA Project Manager	Coordination of project information and related communications on behalf of IPC and MIMC with USEPA; liaison between USEPA project managers and respondent project managers	David Keith	Anchor QEA, LLC 2113 Government Street Building D, Suite 3 Ocean Springs, MS 39564 (228) 818-9626 dkeith@anchorqea.com
Integral Project Manager	Responsible for the successful completion of tasks and coordination with the Anchor QEA project manager, the IPC project manager, and the MIMC project manager to execute the study described in this SAP	Jennifer Sampson	Integral Consulting Inc. 411 1st Avenue South Suite 550 Seattle, WA 98104 (206) 957-0351 jsampson@integral-corp.com
Anchor QEA and Integral Corporate Health and Safety Managers	Oversight of health and safety program for field tasks associated with RI/FS	David Templeton	Anchor QEA, LLC 1423 Third Avenue, Suite 300 Seattle, WA 98101 (206) 287-9130 dtempleton@anchorqea.com
		Eron Dodak	Integral Consulting Inc. 319 SW Washington Street Suite 1150 Portland, OR 97204 (503) 284-5545 edodak@integral-corp.com
Study Elements 1 and 2 Field Lead Integral	Field data collection and implementation of the Health and Safety Plan in the field	Bill Lawrence	Integral Consulting Inc. 411 1st Avenue South Suite 550 Seattle, WA 98104 (206) 230-9600 blawrence@integral-corp.com
Study Elements 3 and 4 Field Lead Anchor QEA	Field data collection and implementation of the Health and Safety Plan in the field for Study Elements 3 and 4	Chris Torell	Anchor QEA, LLC 290 Elwood Davis Road, Suite. 340 Liverpool, NY 13088 (315) 453-9009 x17 ctorell@anchorqea.com

Title	Responsibility	Name	Contact Information
Project Database Administrator	Database development and data management	Dreas Nielsen	Integral Consulting Inc. 411 1st Avenue South Suite 550 Seattle, WA 98104 (206) 957-0311 dnielsen@integral-corp.com
Laboratory QA Coordinator	Completeness of QA documentation and procedures; liaison between project personnel, chemical testing laboratories, and data validators and related QA communications with USEPA	Craig Hutchings	Integral Consulting Inc. 1205 West Bay Dr. NW Olympia, WA 98502 (360) 705-3534 chutchings@integral-corp.com
Laboratory QA Manager	Ensure quality of data; oversee laboratory QA and QC practices, records, and procedures; address nonconformity and corrective actions and reports; and coordinate efforts with laboratory project manager	Julie Gish	Columbia Analytical Laboratory Kelso 1317 S. 13th Avenue Kelso, WA 98626 (360) 577-7222 jgish@caslab.com
		Andrew Biddle	Columbia Analytical Laboratory Houston 19408 Park Row, Suite 320, Houston, TX 77084 (713) 266-1599 abiddle@caslab.com

The following responsibilities apply to the project manager and QA manager at the analytical laboratories used for this task.

The laboratory project manager is responsible for the successful and timely completion of sample analyses, and for performing the following tasks:

- Ensure that samples are received and logged in correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times.
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs).
- Keep the task QA coordinator apprised of the schedule and status of sample analyses and data package preparation.

- Notify the task QA coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met.
- Take appropriate corrective action as necessary.
- Report data and supporting QA information as specified in this QAPP.

The laboratory QA manager is responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program
- Maintain QA records for each laboratory production unit
- Ensure that QA and quality control (QC) procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve nonconformity and corrective action reports
- Coordinate response to any QC issues that affect this project with the laboratory project manager.

1.4 Problem Definition and Background

On March 19, 2008, USEPA added the Site to the National Priorities List (NPL), and the 2009 UAO requires that an RI be conducted at the Site. The investigation described in this SAP will address uncertainties about the following aspects of the Site:

- The nature and extent of Site-related soil contamination
- The exposure to humans and ecological receptors that may be using the Site and may be in direct or indirect contact with contaminated soil
- The physical characteristics of the Site and physical processes governing fate and transport of Site-related contaminated soil.

Relevant background information on the Site, including the Site history and conceptual site model (CSM), can be found in Anchor QEA and Integral (2010b); a description of the Site is provided below. The CSM and Site history presented by Anchor QEA and Integral (2010b) do not address historical waste disposal in areas south of Interstate Highway 10 (I-10), or any related releases of hazardous substances, contaminant transport, or exposure pathways. USEPA is requiring that the investigation include areas south of I-10 and IPC, but not

MIMC, has agreed to perform the investigation in that area. MIMC's position on the southern impoundment is explained in a letter to USEPA from MIMC's legal counsel dated October 21, 2010. This Soil SAP provides all of the details for an investigation of potential soil contamination north of I-10. It also includes a description of the area of interest south of I-10 submitted on behalf of IPC, based on an initial review of available information. This discussion of the southern impoundment does not waive the legal position of MIMC as set out in the aforementioned October 21, 2010, letter. An addendum to this SAP presenting a more detailed historical description of the area south of I-10, a related CSM, and a proposed sampling design was submitted to USEPA on behalf of IPC following additional review of historical records and aerial photographs for this area. Later sections in this SAP describe the uncertainties and data gaps relevant to collection and analysis of new information on soil north of I-10; uncertainties and data gaps associated with soils south of I-10 will be detailed in Soil SAP Addendum 1 submitted on behalf of IPC, after more information has been developed to support a CSM for that area. Additional sections in this SAP describe the sampling procedures, sample custody, analytical procedures, data validation, reporting and management, and QA procedures. Appendix A, the FSP, describes in detail the sampling and data gathering methods, station positioning, field documentation, and all sample handling details. It includes field SOPs and an addendum specific to this study for the project Health and Safety Plan (HASP; Anchor QEA 2009). Soil SAP Addendum 1, submitted on behalf of IPC, includes an FSP Addendum, as appropriate to the sampling stations and methods that will be used for soil sampling south of I-10.

1.4.1 Site Description

The Site consists of impoundments, built in the mid-1960s for disposal of paper mill wastes, and the surrounding areas containing sediments and soils potentially contaminated with the waste materials that had been disposed of in these impoundments. Two impoundments, together approximately 14 acres in size, are located on a 20-acre parcel immediately north of the I-10 Bridge and on the western bank of the San Jacinto River, in Harris County, Texas (Figure 2).

USEPA has identified an area south of I-10 to be investigated, based on historical documents and aerial photographs. These documents indicate that an additional impoundment was

constructed south of I-10, on the peninsula of land directly south of the 20-acre parcel, and was also used as a paper mill waste disposal area in the mid-1960s for paper mill waste similar to that disposed of in the two impoundments. A Texas State Department of Health (TSDH) inspection report dated May 6, 1966, indicates that this older impoundment contained a pond approximately 15 to 20 acres in size (TSDH 1966). Figure 2 shows both the known 1966 perimeter of the impoundments north of I-10 and the potential area of investigation of soils south of I-10. Related uncertainties will be addressed in Soil SAP Addendum 1, submitted on behalf of IPC.

1.4.1.1 Impoundment Locations and Configuration

In 1965, the impoundments north of I-10 were constructed by forming berms within the estuarine marsh, to the west of the main river channel. These impoundments at the Site were divided by a central berm running lengthwise (north to south) through the middle, and were connected with a drain line to allow flow of excess water (including rain water) from the impoundment located to the west of the central berm, into the impoundment located to the east of the central berm (Figure 2).

Review of an aerial photograph from 1964 indicates that an impoundment south of I-10 was also constructed by forming berms adjacent to the shoreline of the peninsula south of I-10 separating the main channel of the San Jacinto River and the Old River. Additional details about the construction and operation of the impoundment south of I-10 are described to the extent possible in Soil SAP Addendum 1. USEPA has provided an interpretation of an aerial photograph from 1964 showing the possible perimeter of the south impoundment (13.4 acres), as well as an interpretation of an historical drawing included in the TSDH (1966) inspection report dated May 6, 1966 (22.8 acres). The larger of these two perimeters was used to define the potential area of investigation shown in Figure 2.

1.4.1.2 Waste Disposal and Waste Characteristics

In 1965 and 1966, pulp and paper mill wastes (both solid and liquid) were reportedly transported by barge from the Champion Paper Inc. paper mill in Pasadena, Texas, and unloaded at the Site into the impoundments, where the waste was stabilized and disposed. The excess water from the impoundments was pumped back into barges and taken off-Site.

The Champion Paper mill used chlorine as a bleaching agent, and the wastes that were deposited in the impoundments have recently been found to be contaminated with dioxins and furans and some metals (TCEQ and USEPA 2006); additional discussion of the chemical constituents typical of materials like those deposited in the impoundments is provided in Section 1.5 of the Sediment SAP for this Site (Integral and Anchor QEA 2010). The impoundments north of I-10 were used for waste disposal from September 1965 through late 1966 until both impoundments were filled to capacity. Because the eastern impoundment north of I-10 was used to dewater the western impoundment north of I-10 (as noted above), the capacity of the eastern impoundment for waste disposal is thought to have been less than that of the western impoundment.

The lateral and vertical extent of any solid wastes remaining in the area, or of soils contaminated by liquid wastes, will be addressed by soil sampling to be described in Soil SAP Addendum 1.

1.4.1.3 Changes Over Time

Physical changes at the Site in the 1970s and 1980s, including regional subsidence of land in the area due to large-scale groundwater extraction and sand mining within the river and marsh to the west of the impoundments, have resulted in partial submergence of the impoundments north of I-10 and in exposure of the contents of these impoundments to surface waters. Historical aerial photography does not indicate that any part of the land south of I-10, or any southern impoundment, has been submerged as a result of subsidence.

Based upon review of U.S. Army Corps of Engineers (USACE)-approved dredging permits, dredging by third parties has occurred in the vicinity of the perimeter berm at the northwest corner of the impoundments that are north of I-10. Recent samples of sediment in nearby waters north and west of these northern impoundments (University of Houston and Parsons 2006) indicate that dioxins and furans are present in nearby sediments at levels higher than levels in background areas nationally (USEPA 2000). Interpretation of historical aerial photographs suggests that the sand mining operation and processing of related sediments extended to the upland area to the west of the northern impoundments, potentially affecting soils in that upland area. Historical aerial photographs also suggest that activities on this

upland area included vehicle traffic and related physical disturbances that resulted in the elimination of vegetation in part of the uplands. On the basis of observable vegetative patterns over time and temporal changes in the locations of objects or material piles on the upland area north of I-10, it appears that handling of sediments on this upland area occurred on the area east of the centerline of the unvegetated uplands, and did not extend to the western half of this uplands area.

1.4.1.4 Surrounding Land Uses

Freshwater, estuarine, and marine habitats occur in the vicinity of the Site. Residential, commercial, industrial, and other land use activities occur within the preliminary Site perimeter and in the surrounding area. Residential development on the eastern bank of the river is present within 0.5 mile of the Site. The impoundments north of I-10 are currently occupied by estuarine riparian vegetation to the west of the central berm, and are consistently submerged even at low tide to the east of the central berm. Estuarine riparian vegetation lines the upland area that runs parallel to I-10 and the uplands west of the impoundments. The impoundment area south of I-10 is currently under industrial or commercial use, including use by a towing company, a shipbuilding company, and a shipyard. Additional uses of this area may have existed in the past and are addressed in Soil SAP Addendum 1, submitted on behalf of IPC. A sandy intertidal zone is present along the shoreline throughout much of the Site (Figure 2).

1.4.2 Summary of Available Soils Data

Both Site and background soils data are relevant to the RI. This section provides a summary of available information on Site and regional background soils.

1.4.2.1 Site Soil Data

At the time the 2009 UAO was issued, there were no chemical data describing soils at the Site, and no physical descriptions of the upland sand separation area to the west of the northern impoundments. Since that time, USEPA has required two soil sampling events to generate information relevant to implementation of the Action Memorandum for a Time Critical Removal Action (TCRA) at the Site, which was issued by USEPA to IPC and MIMC on May 11, 2010 (USEPA 2010a): sampling in the Texas Department of Transportation

(TxDOT) right-of-way (ROW) (August 2010) and sampling on the upland sand separation area to the west of the impoundments north of I-10 (November 2010). Both resulting data sets will be used to address the nature and extent evaluation, the exposure assessments, and the fate and transport evaluation (Study Elements 1, 2, and 3) for the remedial investigation. Provided below are a summary of each and the status of related information. In the discussion below and in Figure 4, dioxin and furan concentrations are expressed as toxic equivalent (TEQ_{DF}) concentrations using mammalian toxic equivalence factors (TEFs) (van den Berg et al. 2006) and assuming non-detects are equal one-half the detection limit for each congener.

Sampling in the TxDOT ROW. Soil samples were collected from 12 locations on the Site at one depth interval (0–12 inches) in 2010 according to specifications in the approved SAP for that sampling effort (Anchor QEA 2010a). These soil samples were analyzed for primary and secondary chemicals of potential concern (COPCs). Validated data in the area of the TxDOT ROW, which lies underneath the I-10 bridge, are available (Figure 4) and have been reported by Anchor QEA (2010b).

A screening analysis of the TxDOT ROW soil chemistry was performed in preparation of the final Soil SAP. Screening values selected for this analysis were the Regional Screening Levels (RSLs) presented by USEPA (USEPA 2010b). RSLs addressing both cancer and noncancer risks for industrial soil were considered, and the lower of the two values for each chemical was selected for screening. For chemicals not addressed by USEPA's RSLs, the Texas Risk Reduction Program Protective Concentration Levels (PCLs) were used (TCEQ 2010). The maximum concentration of each chemical was compared to the screening value. Screening results indicate that concentrations of most chemicals at the majority of stations were either not detected (and had detection limits below the RSL), or were detected and the maximum concentration was below the RSL (Figure 4, Table 1). Exceptions to this were:

- TEQ_{DF} concentrations at two stations immediately adjacent to the impoundments north of I-10 (TxDOT004 and TxDOT005), in the surface 0–12 inch interval. The cancer risk based RSL is 18 ng/kg dry weight (dw), and the concentrations in the surface intervals at these two stations were 66.1 and 20.3 ng/kg, respectively. These values are below other available screening levels for industrial soils, and below USEPA's draft interim soil Preliminary Remediation Goals (PRGs) for residential and for commercial soils (USEPA 2009d).

- Arsenic. All concentrations are below the noncancer RSL. This chemical is common in soil and sediment throughout Texas at levels higher than those measured in these samples. A reference value of 9.61 mg/kg is published by TCEQ (2003) for estuarine sediments, while a state-wide survey of soils in Texas (N = 119) found a range of arsenic concentrations between 1.1 and 18 mg/kg, having a median of 5.9 mg/kg, mean of 6.4 mg/kg, and 95%/95% upper tolerance limit (UTL) of 16 mg/kg (Boerngen and Shacklette 1981).

These results indicate that COPC concentrations in soils from the ROW area are generally low, and with few exceptions are lower than conservative risk screening levels.

Sampling in the upland sand separation area. USEPA required sampling on the upland sand separation area (Figure 3) occurring to the west of the impoundments that are north of I-10, prior to implementation of the TCRA. Analytical chemistry data are available from samples at 13 stations on the upland sand separation area (Figure 4). According to the sampling design, two depth intervals (0–6 inches, or 0–15 cm; and 6–12 inches, or 15–30 cm) at each station were sampled and analyzed for 17 dioxin and furan congeners, total organic carbon, and soil grain size distribution. At a subset of three locations, stations SJTS001, SJTS005 and SJTS009 , soil analytes also included priority pollutant list (PPL) chemicals in both depth intervals (for a total of six samples). Details of the sampling program are presented in an Addendum to the Removal Action Work Plan (RAWP) for the TCRA (Anchor QEA and Integral 2010a).

Results of a screening analysis of these data using the same screening values described for analysis of the soil from the TxDOT ROW (above) indicate that concentrations of most chemicals at the majority of stations were not detected (had detection limits below the RSL), or were detected and the maximum concentration was below the RSL (Figure 4, Table 2). Exceptions to this were:

- TEQ_{DF} concentrations at one station (SJTS010), in both intervals. The cancer risk-based RSL is 18 ng/kg dw, and the concentrations in the surface and subsurface intervals were 27.2 and 26.7 ng/kg, respectively. However, these values are below other available screening levels for industrial soils, and below USEPA's draft interim soil PRGs for residential and for commercial soils (USEPA 2009d).

- Arsenic. All concentrations are below the noncancer RSL. Arsenic is common in soil and sediment throughout Texas. The maximum value detected in soil from the upland sand separation area (2.46 mg/kg dw) was below the reference value, the median concentration in Texas soils, as described above for arsenic in the TxDOT soil samples.

These results indicate that chemical concentrations in soils from much of the area on the upland area to the west of the northern impoundments are generally low, with the exception of one station at which TEQ_{DF} was somewhat greater than the most conservative risk screening level.

Beach sediment samples. Samples were collected in 2010 as a separate component of the RI/FS (Integral and Anchor QEA 2010). These beach sediment samples were collected from the eastern and western shorelines of the property west of the impoundments, and at locations between the impoundments and this upland property. Beach sediments were collected from 0 to 6-inch and 6 to 12-inch depth intervals between the high tide mark and the low tide mark. These samples have also been analyzed, validated, and provided to USEPA in a database update on September 18, 2010. For the stations located along the eastern shoreline of the upland sand separation area (Figure 3, Area 1)TEQ_{DF} concentrations ranged from 0.316 to 10.9 ng/kg, having values less than 3.0 ng/kg for all but one of the sampled stations. These results indicate that TEQ_{DF} concentrations along the eastern shoreline of Area 1 are below the cancer risk-based RSL for soil of 18 ng/kg dw.

1.4.2.2 Background Soil Data

Soil samples from the Houston area were collected by the Total Maximum Daily Load (TMDL) program (University of Houston and Parsons 2006). To evaluate the physical and chemical processes that facilitate the transfer of soil contaminants into the aquatic environment through surface water transport, soils were collected for analysis of organic carbon and dioxins and furans from one or more of five land use types (forest, grass, residential, transitional, and urban) concurrently with runoff at ten different locations across the city of Houston (Table 3). Soil samples were collected between December 2004 and October 2005; the depth of soil samples was not specified. Grain size distribution was

also measured during runoff sampling. A review of the laboratory quality assurance information for these data is under way; the data discussed below and in Table 3 were taken from the project database, and may differ from those reported in the TMDL reports (see Section 3.3. of the RI/FS Work Plan).

Percent organic carbon ranged from 0.88 to 5.70 (forest soil), 1.47 to 4.87 (grass soil), 0.90 to 5.09 (residential soil), 0.28 to 2.53 (transitional soil), and 1.43 to 5.66 (urban soil) across all ten locations during runoff sampling. TEQ_{DF} concentrations in soils across the ten locations (Table 3) (ng/kg dry weight) ranged from 0.57 to 8.9 (soil), 0.36 to 8.0 (forest soil), 0.46 to 7.7 (grass soil), 0.42 to 5.0 (residential soil), 0.39 to 28 (transitional soil), and 0.46 to 2.28 (urban soil). These values are consistent with and generally lower than TEQ_{DF} concentrations measured in an extensive urban background study in residential, commercial, and industrial areas from Denver, Colorado (USEPA 2001b).

1.4.3 Problem Definition

Contact with contaminated soil in areas to which waste materials and contaminated sediments from the northern impoundments may have been transported creates the potential for exposure of ecological receptors and people using the Site to COPCs. Ecological receptors and people using the Site also may be exposed to COPCs from global, regional, and local sources that are unrelated to the Site. The overall CSM (Figure 5), and exposure CSMs for human and ecological receptors relating to the impoundments north of I-10, are illustrated and discussed in greater detail in the RI/FS Work Plan (Anchor QEA and Integral 2010b). A CSM addressing potential transport and exposure pathways for the impoundment south of I-10 are presented in Soil SAP Addendum 1, submitted on behalf of IPC.

Dioxins and furans are known to be present in sediments within and around the impoundments that are north of I-10; related data and analyses are summarized by Anchor QEA and Integral (2010b). Because of activities associated with the sand mining and related handling of sediments that took place in the upland area west of the impoundments north of I-10, it is possible that transfer of sediment contaminants to upland soils has occurred. The nature and extent of potential contamination of soils across the Site with COPCs from the impoundments is unknown. The potential for exposures of human and ecological receptors

resulting from contact with soils is also unknown. Finally, if surface soils are contaminated in upland areas, it will be necessary to evaluate the potential for transport from uplands into the water, which could create the potential to contaminate sediments, or recontaminate sediment following sediment remediation.

The overarching issue to be addressed by the study described in this SAP is whether chemicals associated with sediments or wastes in the impoundments occur in soils in the upland areas, including those west of the impoundments and on the peninsula south of I-10, and if so, the nature and extent of their distribution in affected soils. Resulting data will be used to evaluate exposures and risks to ecological receptors and people. Both the exposure and risk assessment, and characterization of background conditions in soil will inform the development of PRGs, if evaluation of remedial actions for soils is determined to be necessary. Data resulting from the nature and extent evaluation in combination with additional information on the microtopography of the uplands that may have been affected by wastes, are needed to determine possible surface transport pathways that could transfer COPCs in soil to the aquatic environment. Where groundwater wells will be installed for evaluation of groundwater quality (Groundwater SAP; Anchor QEA 2010c), the chemistry, grain size, and lithology of soils from the well borings may be needed to facilitate interpretation of groundwater data.

1.5 Chemicals of Potential Concern and Soil Analytes

This section discusses the selection of soil analytes. However, there are different uncertainties for soils north of I-10 than for soils south of I-10. Therefore, the process for COPC identification for soils differs between the two areas. The Sediment SAP (Integral and Anchor QEA 2010) describes the process and rationale for selection of primary COPCs and secondary COPCs for media that may have been contaminated by wastes deposited in the northern impoundments, and how their analyses relate to those for the indicator chemical group, dioxins and furans (this discussion is also provided as Appendix C to the RI/FS Work Plan; Anchor QEA and Integral 2010b). A more detailed discussion will be presented in the forthcoming COPC Technical Memorandum (in response to USEPA comments on the Tissue SAP) for the northern impoundments.

Consistent with these documents, dioxins and furans will be measured in soils from all areas of the Site (Table 4), as will other primary COPCs (Table 5). For soils to be collected in areas north of I-10, additional analytes among the secondary COPCs will be identified through analyses of sediment data, as follows. Results of sediment chemical analyses from the sediment sampling conducted in May 2010 will be generated prior to the performance of soil sampling. Using validated chemistry data for sediments, results for secondary COPCs will be evaluated 1) for frequency of detection in sediments, 2) against risk-based screens, and 3) for statistical correlation with dioxins and furans in sediment that are representative of the wastes in the impoundments north of I-10 (e.g., one or more of the most common congeners in waste-related sediments). These evaluations will result in one of the following outcomes:

- Those secondary COPCs detected in fewer than 5 percent of sediment samples will not be considered in the risk assessments, and will therefore not be measured in soil.
- Secondary COPCs detected in sediment will be evaluated using the risk-based screening procedures established by the Sediment SAP. If a COPC passes the risk-based screen and is not bioaccumulative, it will not be considered further. Chemicals that exceed screening values (for soils), or that have the potential to bioaccumulate, will be evaluated for correlation with dioxins and furans considered representative of the wastes in the impoundments.
- Secondary COPCs that are detected in sediments, that fail the risk-based screens, and that statistically correlate with representative dioxin and furan congeners will not be evaluated in soil, unless additional information indicates that the risks should be evaluated for the chemical. A correlation with dioxins and furans, the chemicals that are likely the primary risk drivers, will be interpreted to indicate that remedial actions to address dioxins and furans will also address any risks due to secondary COPCs.
- Secondary COPCs that are detected in sediments that fail the risk-based screens and that do not statistically correlate with representative dioxin and furan congeners will be evaluated in soil.

As noted for sediment and tissue COPCs, these decision rules apply unless additional information indicates that a COPC may or may not be present at elevated levels in soil on Site as a result of activities associated with sediments that may have been affected by waste from the impoundments that are north of I-10. Information that may indicate a COPC is not

expected at elevated levels in soils includes information on the persistence of a chemical in a terrestrial environment. The COPC Technical Memorandum will document analyses and decision points to address all of the primary and secondary COPCs, and their role in the risk assessments and other future analyses for the RI/FS.

Therefore, the initial analytes in soils collected north of I-10 for this study are the primary COPCs (Table 5). For all secondary COPCs, analytical methods, mass requirements, holding requirements, and other QA/QC procedures are completely described in this SAP, so that they can be effectively analyzed in soil, as appropriate to the findings of the sediment study. Primary COPCs will be analyzed immediately after collection of soils, and soils will be archived for analysis of secondary COPCs.

For soils south of I-10, the following information was considered in identification of soil analytes and is being submitted on behalf of IPC. Because the materials deposited in the impoundments south of I-10 may be of the same origin and types as those deposited in the northern impoundments (TSDH 1966), analytes for soils collected south of I-10 were determined in the same manner as the analytes for sediments in the northern impoundments, i.e., through the analysis and considerations detailed in Appendix C to the RI/FS Work Plan (Anchor QEA and Integral 2010b), and in Section 1.5 of the approved Sediment SAP (Integral and Anchor QEA 2010). For soils to be collected south of I-10, initial analytes will include all of the chemicals of interest (COIs) identified in the Sediment SAP (Integral and Anchor QEA 2010) and listed in Table 5 of that document, modified to include all of USEPA's target analyte list (TAL) metals. COIs represent those chemicals that are:

- On the target analyte list for metals, the priority pollutant list for surface water, and on the contract laboratory program analyte list,
  - and are potentially associated with pulp mill solid wastes or effluents,
  - and are persistent in the environment,
- or were detected at least once in samples collected by TCEQ and USEPA (2006) (see Section 1.5 of the Sediment SAP)

There are no pre-existing samples for soils in the south impoundment that allow performance of a risk-based screen. Given that the origin and types of the waste are believed to have been the same as for the material deposited in the impoundments north of I-10, this

approach relies on the same logic as described in Section 1.5 of the Sediment SAP. It is conservative in including all chemicals that were considered prior to the screening step, and all target analyte list (TAL) metals that were eliminated on the basis of an evaluation of waste characteristics. The TAL metals that have been added to the COI list in Soil SAP Addendum 1 are beryllium, iron, and selenium[1]..

Additional analytes in all samples will include total organic carbon and grain size.

1.6 Uncertainties and Data Gaps

Uncertainties and data gaps for soils on the Site are discussed below. The soil study proposed in this document addresses the collection and analysis of new information to address and reduce the uncertainties concerning the nature and extent of contamination, exposure potential and risks due to contamination of soils with wastes or sediment associated with the northern impoundments, and potential for post-remediation recontamination of sediment as a result of soil transport.

1.6.1 Nature and Extent

There are currently no data to describe the chemistry of soils on the Site (except as described in Section 1.4), but the Site history and CSM suggest that sediments from within the northern impoundments may have been transferred to the sand-sorting area of the upland portion of the property west of the impoundments. Site history also suggests that the extent of sediment handling in the uplands was greatest on the eastern half of the unvegetated upland area west of the northern impoundments. Appropriate soil data for characterization of the nature and extent of contamination in the areas on this upland that is north of I-10 that may have been affected by dioxins and furans in sediments represents a data gap. This data gap will be addressed by collecting soil data in the upland area north of I-10 using a sampling design that will produce accurate and representative estimates of dioxin and furan concentrations in surface soil.

[1] Potassium and sodium are TAL metals, but will not be addressed as COIs because they are natural soil constituents, are physiologically regulated by reptiles, birds and mammals and readily excreted, do not bioaccumulate, and are therefore assumed to be very unlikely to be risk drivers in soil.

There are also no soil chemistry data for the impoundment area south of I-10. Appropriate surface and subsurface data for characterization of nature and extent of contamination represents a data gap. Data gaps for the soil south of I-10 are discussed in greater detail in Soil SAP Addendum 1, submitted on behalf of IPC.

In addition, the CSM identifies global, regional, and local atmospheric activities and emissions as potential sources of dioxins and furans to the Site, including to soils on the Site. Therefore, to understand the nature and extent of soil contamination potentially linked to the impoundments, characterization of dioxins and furans in soils from background areas are required. Data describing dioxins and furans in background soils are not currently present in the database, and therefore concentrations of dioxins and furans in background soil are a data gap.

Finally, ancillary information required to interpret soil chemistry data (e.g., in comparisons between samples or between areas) include the total organic carbon (TOC) content of soils, and the grain size distribution.

1.6.2 Human and Ecological Exposures

Human and ecological receptors may be exposed to contaminated soils in the upland area west of the northern impoundments. Four types of human receptors have been identified in the RI/FS Work Plan for the baseline human health risk assessment: subsistence fisher, recreational fisher, trespasser, and recreational user. According to the CSM for human health presented in the RI/FS Work Plan, fishers and recreational visitors have potentially complete and significant soil exposure pathways via direct contact, which includes incidental ingestion and dermal contact, while for trespassers, direct contact is potentially complete but minor. Inhalation of airborne dust is also a relevant exposure pathway for human receptors. The ecological exposure CSM indicates that there are no complete exposure pathways to soil for benthic invertebrates and fish. Ingestion of soils is a complete and significant pathway for reptiles, birds, and mammals, and direct contact exposure to these receptors is considered potentially complete but minor. Because there are currently no data to describe dioxins and furans in soils on the Site, information required to evaluate baseline exposures of human and ecological receptors coming into contact with those surface soils in the upland area west of

the impoundments is needed. Information is also required to evaluate baseline exposures of human and ecological receptors coming into contact with surface and near surface contaminated soils within the impoundment north of I-10.

A CSM for the southern impoundment is derived and presented in Soil SAP Addendum 1, submitted on behalf of IPC. Because of land uses at the location of this impoundment, an additional human receptor group, the industrial worker, will be considered in the risk assessment. In addition, it may not be necessary to address baseline risks to recreational and transient users, or children, if these receptors do not have access to areas with potentially contaminated soils. Data gaps specific to the industrial worker and ecological receptors are addressed in Soil SAP Addendum 1,

As for the nature and extent evaluation, characterization of soil-related exposures to dioxins and furans potentially attributable to the waste stored in the impoundments requires information on the soil-related exposures of dioxins and furans from background areas. Information on dioxins and furans in background soil is also considered a data gap.

1.6.3 Physical CSM and Fate and Transport Evaluation

Because sediments potentially contaminated with dioxins and furans may have been transferred to uplands north of I-10, processes of erosion could transfer dioxin and furan-contaminated soils back into the aquatic environment, potentially contaminating surface water and sediments adjacent to the uplands. For the evaluation of remedial alternatives, information on the potential transfer pathways for dioxins and furans from uplands to the aquatic environment is needed. Concentrations of dioxins and furans in soils, and the physical transfer pathways for potentially contaminated soils to the aquatic environment will be required to characterize the extent of potential transfer of COPCs via erosion, and the spatial distribution of areas where soil deposition could affect sediment quality. Therefore, in addition to data gaps for dioxins and furans in soils in the upland areas west of the impoundments and south of I-10, the specific hydrological pathways that could facilitate the transfer of soils into the aquatic environment via surface runoff are unknown. Information on the topography of the upland portion of the property west of the impoundments at a resolution appropriate to identification of potential surface transport pathways is a data gap.

An additional data gap relating to this Study Element is data for the chemistry of soils within borings of three groundwater monitoring well pairs that will be installed north of I-10 to evaluate the chemistry of groundwater. Anchor QEA's Groundwater SAP (Anchor QEA 2010c) describes a groundwater sampling program that will be used to determine if COPCs from the Site are present in groundwater underneath the impoundments north of I-10. Soil lithology, grain size distribution, and chemistry data from the locations where the wells are drilled may be needed to interpret the groundwater data. Data gaps for soils relating to the evaluation of groundwater in areas south of I-10 are addressed in Soil SAP Addendum 1, submitted on behalf of IPC.

1.6.4 Engineering Design Evaluation

Until the nature and extent, potential for exposure, and potential for surface transport are better characterized, data gaps relating to engineering design cannot be defined. Any need for additional soil data relating to an engineering design evaluation will be addressed in an addendum to this SAP.

1.7 Task Description

The soil study will address data gaps by generating new data for soil chemistry at the Site. It consists of a series of tasks, to be executed by Integral and Anchor QEA on behalf of respondents, and in consultation with USEPA:

- Agreement on the study design and finalization of a complete Soil SAP and Soil SAP Addendum 1 for the area south of I-10
- Fieldwork to collect the required soil samples, and appropriate execution of contingency plans as needed for conditions in the field
- Effective communication of modifications to the SAP, a consensus view of the means to address required changes, and employment of contingencies and alternatives identified during the field sampling
- Effective processing, handling, shipment, and analyses of soil samples, all of which conform to specifications of this SAP

- Complete documentation of sample collection, deviations from the SAP, field activities and observations, sample processing and shipping, chain of custody requirements, and analytical procedures
- Validation of soil chemistry and conventionals (organic carbon and grain size) data according to specifications in this SAP
- Complete and timely loading of validated data into the project database, and dissemination of the data to USEPA and interested parties.

The soil study will address data gaps by generating new information relating to three of the four study elements that have been defined for the RI/FS (Anchor QEA and Integral 2010b):

- **Study Element 1: Nature and Extent Evaluation**. Data will be used to characterize the nature and extent of COPCs in soils in the uplands west of the northern impoundments and in soils potentially affected by waste handling in areas south of I-10.
- **Study Element 2: Exposure Evaluation**. Data will be used to evaluate the potential ecological and human exposures and related health risks resulting from contamination of upland soils on the property west of the northern impoundments, within the northern impoundments, and in soils potentially affected by waste handling in areas south of I-10.
- **Study Element 3: Fate and Transport Evaluation**. Data will be used to identify physical transport pathways and to evaluate the potential for transport of COPCs in soil to the aquatic environment. Soil chemistry, lithology, and grain size data may be needed to evaluate the potential for transport of COPCs in soil to groundwater within the Site.

Completion of Study Elements 1 through 3 (as described in this document) will allow evaluation of the nature and extent of contamination of soils with COPCs, determination of whether COPCs in soils are associated with unacceptable risks to human and ecological receptors, and determination of whether COPCs may be transferred from the uplands areas to the aquatic environment. After these evaluations are complete, a decision will be made to determine if remediation of soils is required, and if so, whether soil data generated by this study are sufficient to support design of remedial actions. If additional sampling is required,

then an addendum to this document will be prepared to describe the approach and requirements of Study Element 4: Engineering Construction Evaluation.

Data Quality Objectives (DQOs) for each element as they pertain to the impoundments north of I-10 and related soil contamination are discussed in Section 1.8. The study design for the northern part of the Site is described in greater detail in Section 2.1. DQOs and study design details specifically for soil sampling south of I-10 will be presented in Soil SAP Addendum 1, submitted on behalf of IPC. Generally, DQOs for the south impoundments will directly mirror those shown below. Analytes for all soil samples for the exposure evaluation include primary COPCs, with collection and archiving of soils for potential future analysis of secondary COPCs (Table 5), if required by the process described in Section 1.5.

Sampling of soil will take place in early 2011 (Anchor QEA and Integral 2010a), unless other agreements regarding the sampling period are made in consultation with USEPA.

1.8 Data Quality Objectives

This section presents a summary of the DQOs for soil sampling to evaluate nature and extent, human health and ecological exposure assessments, and the potential physical transport pathways of soils to the aquatic environment or to groundwater north of I-10. DQO discussions for Study Elements 1 and 2 are combined because the sampling objectives and analysis plans for these two study elements are integrated for soils. These DQOs have been prepared consistent with USEPA guidance (USEPA 2006). Establishing DQOs assures that data generation and sampling will be focused on the goals of the RI/FS and will be sufficient to address those goals. The DQO summaries in the following subsections include, for each study element, a statement of the problem, components of the sampling design necessary to support the analytical or interpretive approach, and a description of the analytical approach to be followed.

1.8.1 DQOs for Study Elements 1 and 2: Nature and Extent Evaluation and Exposure Assessment

The RI/FS is being undertaken to address contamination of San Jacinto River sediments within and in the vicinity of the impoundments at the Site (Figure 2), and to address

contamination of other environmental media that have been in contact with the waste or contaminated sediments in or associated with the impoundments. The soils on the uplands to the west of the impoundments may have been affected by COPCs from the impoundments. To effectively plan for any remedial actions that might be required, the spatial and vertical extent of soil contamination will be evaluated, at least in part, by comparison of soil data to concentration-based PRGs for soils.

The RI/FS will address risks to human and ecological receptors associated with contamination of Site soil that may have resulted from activities on the uplands west of the impoundments related to handling of sediments at the Site, and risks associated with soil contamination within the impoundments north of I-10. The exposure evaluation and risk assessment will support planning for remedial actions. This section presents the technical rationale and general approach for conducting the evaluation of human and ecological exposures to COPCs in soil from the Site.

1.8.1.1 Statement of the Problem

Problems relating to characterization of the nature and extent of contamination, and to the exposure assessment will be addressed by this study.

1.8.1.1.1 Nature and Extent

The primary problem to be addressed by Study Element 1 of the RI/FS (the nature and extent investigation) is uncertainty in the spatial and vertical extent of contamination in soils on the upland area to the west of the impoundments. A related problem to be addressed by Study Element 1 is that comparison of COPCs in Site soils with COPCs in soils from background areas is needed to evaluate the relative contribution of wastes or sediments from the impoundments to any COPCs identified in soil. The nature and extent evaluation, including characterization of soils in comparable background areas, will address these problems and thereby facilitate the selection and implementation of remedial approaches, if required.

Evaluation of the importance of Site-related COPCs in upland soils relative to atmospheric, global, and other sources requires characterization of contaminated soils using dioxin and furan signatures. Upland areas in the vicinity of the Site and potentially subject to the same

types of regional and atmospheric influences as soils at the Site (e.g., traffic on freeways) are relevant for assessing soil conditions and soil chemistry that could occur as a result of processes other than those that may have transferred materials from the impoundment to the uplands west of the impoundments. Although some soils data for urban, residential, forested, grassy, and transitional areas in the Houston area have been previously collected (Table 3), a larger number of samples is required for quantitative comparison (Gonzales 2007). Because of the potential influence of traffic on rates of atmospheric deposition of dioxins and furans (University of Houston and Parsons 2006), and the proximity of the upland area west of the impoundments to I-10, background areas selected for collection of soils and comparison to the Site soils should be similar to this Site in terms of proximity to traffic. Samples from background areas that are as close to the freeway as the Site are needed to ensure that the influence of background sources on Site soils is characterized.

1.8.1.1.2 Exposure Assessment

People visiting this portion of the San Jacinto River may be exposed to COPCs in soil via direct contact (ingestion and dermal) with soils or inhalation of airborne particulates that may have been affected by handling of contaminated sediments in uplands west of the impoundments (Figure 5) and via contact with soils that are within the impoundments or on the impoundment perimeter berms. Characterization of risk in support of selection and implementation of remedial approaches requires information on contamination in soils accessible to people. One problem to be addressed by the soil study is uncertainty and data gaps regarding concentrations of primary COPCs present in soil directly contacted by people visiting the Site.

A related problem is the potential for ecological receptors at the Site to be exposed through direct ingestion of contaminated soil. The problem to be addressed in the ecological exposure evaluation is uncertainty regarding the magnitude and spatial extent of exposures of birds, mammals, and reptiles to contaminants in Site soils.

For both human and ecological receptors, there is additional uncertainty regarding the exposures to COPCs in soils of background areas. Information on exposures and risks to

human and ecological receptors both at the Site and in background areas are needed in the evaluation of remedial options.

1.8.1.2 Sample Collection Design

The soil sampling design for Study Elements 1 and 2 was developed in consideration of the following:

- Designation of areas for soil collection on the basis of the extent to which an area could have been affected by handling of sediments
- Spatial distribution of sampling stations
- Depth of COPC contamination and depth at which human and ecological exposures may occur
- Total sample numbers necessary for exposure assessment
- Characterization of background in areas generally equivalent to the Site in terms of non-Site influences.

Soil Collection Areas

To evaluate the nature and extent of contamination, four areas were defined (Figure 3): Area 1 is the unvegetated portion of the upland area west of the impoundments, where historical aerial photographs suggest the majority of sediment handling took place, and the area surrounding the road that provides access in and out of the upland area;. Area 2 is the portion of the Site beneath I-10, and was sampled for the TCRA (Anchor QEA 2010a). Area 3 is the area of the impoundments north of I-10, and Area 4 is the area of soil investigation that is south of I-10.

Spatial Distribution of Site Samples

To characterize the nature and extent of impoundment-related contamination in upland soils to the west of the impoundments north of I-10, soil samples will be collected from an approximately regular grid, with some locations targeting areas of potential contamination (Figure 3). Sample placement and spacing has been chosen to address the data gaps on areas not covered by the results of the available soils data presented in Section 1.4.2.1. The grid is guided by 140-foot (43-m) spacing in Area 1 and targeted to the portions not covered by the existing TCRA dataset (Anchor QEA and Integral 2010a). Additional samples will be

collected along the road leading away from the upland area west of the impoundments on the northern road shoulder, adjacent to the path of trucks and other vehicles exiting the upland sand separation area (Figure 3).

Area 2 has already been sampled and results presented by Anchor QEA (2010b); no additional sampling will be conducted.

Samples in Area 3 have been collected in accordance with this SAP at the stations west of the central berm in the impoundments north of I-10, and at the locations of six geotechnical cores; samples at the locations of the three paired groundwater wells will be collected when groundwater wells are drilled, in late December, 2010.

Samples in Area 4 will be distributed in the area of investigation and collected as described in Soil SAP Addendum 1.

Sample Depth and Analytes

In Area 1, surface and shallow subsurface soil samples will be collected at two depths, 0–6 inches (0–15 cm), 6–12 inches (15–30 cm), respectively. Cores (12–24 inches [30–60 cm]) for nature and extent characterization will be collected at 8 stations in Area 1. In Area 3, surface and shallow subsurface samples have been or will be collected at nine locations with depth intervals of 0–6 inches (0–15 cm) and 6–12 inches (15–30 cm). Within the impoundments north of I-10, cores have been collected as part of the sediment study (Integral and Anchor QEA 2010). All samples will be analyzed for primary COPCs, TOC, and grain size. In the background area(s), samples will be collected at 0 to 6 inches (0–15 cm) and 6–12 inches (15–30 cm) only and analyzed for primary COPCs, TOC, and grain size.

At all sample locations on the Site, and for all depth intervals, sufficient mass of soil will be collected and archived for potential analysis of secondary COPCs.

Number of Samples

The overall design produces samples at 52 locations (including the 25 locations sampled for the TCRA) on the Site (additional locations will be sampled south of I-10, as described in SAP Addendum 1) with high spatial density of samples in areas most likely affected by

sediment handling in the uplands north of I-10. Table 6 provides a summary of the numbers of locations in each area, but does not include the TCRA samples (Section 1.4.). The total sample size for Area 1 resulting from all sampling (i.e., 31 locations) will exceed the requirements for calculation of an upper confidence limit (UCL) for the human health risk assessment for this area. In Area 3, nine locations will be sampled within the impoundments north of I-10 or on its berms for evaluation of exposure (note that there are more than a dozen surface sediment samples of various depths within or near those impoundments).

Background Conditions

Surface soils in background areas will be collected to allow comparison of soil samples from within the preliminary perimeter to local background conditions as part of the nature and extent investigation. A total of 20 background stations will be sampled in one or more of the locations depicted in Figure 6; specific locations for sampling will depend on accessibility, safety, and permissions to sample soils. Information on the use of soil amendments, including those with chemical additives or pre-existing contamination, will be assembled before sampling in the locations depicted in Figure 6. If such information indicates that the areas shown in Figure 6 are potentially inappropriate as background areas, additional areas will be identified for background sampling, in collaboration with EPA.

Surface soils and the shallow subsurface soils will be sampled at the same depth intervals as for those at the Site: 0–6 inches (0–15 cm) and 6–12 inches (15–30 cm); soils will not be collected from background areas at 12–24 inches (30–60 cm). A minimum of 20 total samples is necessary to accurately calculate statistics to represent background (such as the upper 95th percentile of the background data, or a 95 percent upper prediction limit, which is used by the Texas Commission on Environmental Quality [TCEQ] for characterizing background), and therefore will be collected from acceptable background areas for this study. The 10 existing samples for soils in residential and urban areas from the TMDL program (Section 1.4.2.2) can not be evaluated for data quality because appropriate laboratory records are not available.

1.8.1.3 Analytic Approach

Study Element 1 includes the following distinct types of analyses for Area 1:

- **Characterization of the spatial extent of contamination.** Analysis of the samples in Area 1 will be conducted according to the following steps.
  - Area 1: To characterize the nature and extent of contamination in soils, the 0–6 inch (0–15 cm) and 6–12 inch (15–30 cm) depths will be analyzed immediately after sampling at all stations; and the 12–24 inch (30–60 cm) interval at all locations (8 stations). Concentrations of primary COPCs will be quantified in all samples.
- **Surface soil data will be evaluated to determine whether there are distinct areas of elevated contamination or distinct spatial gradients within Area 1.** This evaluation will be carried out using geostatistical methods such as kriging (Myers 1997), and multivariate statistical tools such as principal components analysis (PCA), similarity metrics, and unmixing analysis (Unmixing); a comprehensive review of these methods can be found in Johnson et al. (2007). A summary of unmixing methods is provided in Appendix C. Indexes of risk assessment results (e.g., locations with risk higher than specified thresholds) may be mapped to support the visualization and interpretation of risk assessment results.
- **Evaluation of the association of contaminants in soils in the upland area west of the impoundments with the contaminated materials within in the impoundments.** Patterns of dioxin and furan congeners within a soil sample can vary considerably depending on the source (USEPA 2004). Therefore, a pattern-matching approach using data for dioxins and furans will be used to evaluate sediments or wastes from within the impoundments, and both Site and background soil samples to identify any pattern characteristic of the impoundment. Results will be used to determine the contribution of sediments with this pattern to soils in the upland area west of the impoundments, and in the vicinity of I-10. The pattern-matching approach (such as PCA or Unmixing) will provide an estimate of the fractional contribution of different source types to each soil sample. Assuming that the results of pattern-matching analyses can be interpreted as sources, this analysis will provide the basis for determining the fractional contribution of the impoundment to any dioxin or furan contamination of each soil sample. A broad discussion of the methods for this

pattern-matching approach is presented in Section 6.1.7 of the RI/FS Work Plan (Anchor QEA and Integral 2010b) and additional details on the history and use of Unmixing is provided as Appendix C.

- **Comparison of Site soil conditions with background soils.** Evaluation of Site data relative to background conditions requires assessment of variability in background conditions. For this analysis, samples will be collected in the surface and shallow subsurface intervals 0–6 inches [0–15 cm] and 6–12 inches [15–30 cm]) in one or more background areas, and comparisons with Site data will be made. Consistent with USEPA guidance for evaluation of background soils (USEPA 2002c), an upper 95th percentile or upper tolerance limit (UTL) will be derived to characterize background conditions. This approach provides a threshold value for comparing individual Site stations to background conditions. Background areas are those with public access and that are directly adjacent to or very near high-traffic roadways, where atmospheric dioxin and furan sources are likely comparable to those at the Site (Figure 6). TCEQ prefers the use of an upper prediction limit (UPL) for background comparisons, so this statistic will also be calculated and considered.

Study Element 2 will include the following types of analyses:

- **Characterization of exposures to human receptors using the Site.** Sampling of soils for Study Element 1 will provide data that are useful for evaluating exposure of people to surface soils, at both the 0–6 inch (0–15 cm) and the 6–12 inch (15–30 cm) depth intervals. A total of 31 locations will be sampled and analyzed in Area 1, 12 locations have been sampled in Area 2, and 9 locations have been sampled in Area 3. The data from these samples will be used to calculate exposure point concentrations to represent the central tendency (CT) and reasonable maximum exposures (RME) of each primary COPC in soil for use in the risk assessments.
- **Comparison of exposures of human and ecological receptors to COPCs in Site soils to those of background.** Exposures to soil contaminants on the Site will be compared with exposures at background locations (Figure 6) to determine the extent to which Site soils pose an excess risk to people, reptiles, and birds. Sampling of soils in background areas for Study Element 1 will provide the necessary data for evaluation of background exposures.

1.8.2 DQOs for Study Element 3: Physical CSM and Fate and Transport Evaluation

The RI/FS will provide information to characterize the potential movement of impoundment-associated contaminants in soils from uplands back into the aquatic environment as a result of surface erosion. This information is necessary to determine whether soils could contribute to sediment contamination, and thereby to evaluate whether remedial actions are needed.

The RI/FS will also include a limited study of groundwater involving the installation of three groundwater monitoring well pairs in the vicinity of the impoundments. A complete SAP for collection and analyses of groundwater has been submitted to USEPA (Anchor QEA 2010c). During well installation, soil samples will be collected from the boreholes required to install the deeper of each of the well pairs, and resulting soil data will be used to interpret subsurface geology, and may be used to interpret groundwater chemistry results.

1.8.2.1 Statement of the Problem

The goal of Study Element 3 of the RI/FS is to determine primary physical and chemical processes controlling chemical fate and transport, and to use that information to refine the CSM for the Site. The problems to be addressed by the soil study pertain to:

- The topographical conditions of the uplands west of the impoundments or on the impoundments south of I-10 that could facilitate transport of COPC-contaminated soils from uplands to the aquatic environment
- The geological or chemical conditions that could result in contamination of groundwater with COPCs.

1.8.2.1.1 Topography of the Uplands

On the upland areas, if soils are contaminated with COPCs originating from the impoundments, surface water runoff could erode soils back into the aquatic environment. The topography of the uplands area within which soils will be sampled will determine the physical transport pathways that exist for the movement of soils to the aquatic environment.

1.8.2.1.2 Soil Quality at Groundwater Well Locations

An additional problem relating to the understanding of fate and transport of COPCs on the Site is uncertainty about the subsurface geology and the potential for COPCs to enter the shallow groundwater and the aquifer. Additional information on soil lithology and soil grain size at groundwater well locations is needed to address these uncertainties. Additional chemistry data may be needed to interpret results of groundwater sampling.

1.8.2.2 Sample Collection Design

The sampling design for Study Element 3 was developed in consideration of the following:

- The spatial and vertical resolution required to effectively describe possible surface water transport pathways on the uplands west of the impoundments
- The spatial distribution of groundwater wells.

Sampling to address Study Element 3 in the area south of I-10 will be conducted in consideration of these same factors, and is discussed in Soil SAP Addendum 1.

Surface Topography

Light detection and ranging (LiDAR) data developed in 2008 and describing the surface topography of the Site at a resolution appropriate for developing surface flow paths will be purchased from the Houston-Galveston Area Council. Both vendor-provided surface descriptions (such as 1-foot contour lines) and the bare-earth and all-return point data will be obtained and used to interpret the topography of the uplands west of the impoundments. Data will be interpreted using geographic information system software (ArcGIS) to interpolate a digital elevation model from the bare-earth point-return data and to perform an analysis of hydrologic flow paths. The digital elevation model will represent surface topography of the upland area west of the impoundments in 1-foot pixels, with a vertical accuracy of 0.22 foot. No field activities will be required.

Soils at Groundwater Well Locations

Three pairs of boreholes (one "shallow" and one "deep" in each pair) will be advanced in locations on the Site to enable the groundwater monitoring well pair installation, as described in the Groundwater SAP (Anchor QEA 2010c). Soil samples will be collected from

the deeper of the two borings during the process of establishing groundwater monitoring wells at multiple depth intervals at a given station. Observations on soil lithology (color, grain size, consistency, etc.) will be recorded following visual examination during drilling and sampling activities; these soil samples will be inspected and logged in accordance with American Society for Testing and Materials (ASTM) D2488 *Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)*. Soil samples will be collected for grain size analysis from 0 to 5 feet below grade, and at every 5-foot interval thereafter (10 to 11 feet, 15 to 16 feet, etc.), with soils across the entire depth interval to be composited for grain size analysis and chemistry. Grain size analyses will be conducted immediately after sampling. Sufficient mass of soil for analysis of primary and secondary COPCs from each of the 5-foot intervals will also be collected; these samples will be archived for possible future analysis, depending on the findings of the groundwater sampling.

1.8.2.3 Analytic Approach

The analysis of data to be collected for Study Element 3 includes development of hydrologic flow paths models on the surface of the upland areas west of the impoundments north of I-10, and in the area where impoundments are suspected to have occurred south of I-10, and use in interpretation of groundwater sampling results.

Surface Topography

The ArcHydro extension in the ArcGIS software package will be used to delineate surface drainage flow paths of site topography. The 1-foot bare-earth digital elevation model grid will be used as input to produce a flow direction grid, in which grid cells indicate the flow direction defined by slope calculations using an eight direction pour point model. The flow direction grid will be used as input to produce a flow accumulation grid, which records the number of cells that drain to a specific cell in the grid. Flow paths will be defined from the flow accumulation grid with the use of threshold drainage areas. Flow accumulation grid cells greater than the threshold drainage area will be classified as flow paths and all cells less than the threshold will be interpreted as areas contributing to the flow paths. The resulting flow paths will identify dominant drainage flow patterns on the upland area.

Soils at Groundwater Well Locations

The Groundwater SAP (Anchor QEA 2010c) describes the analysis of groundwater chemistry. Soil lithography at each of the groundwater well locations will be used in the evaluation of possible transport pathways from surface to groundwater, if groundwater quality is found to potentially be affected by surface conditions. Soil samples archived for possible chemical analyses will be analyzed if information on soil and sediment chemistry produced as a result of the soil data developed for the TCRA (Anchor QEA 2010a), Study Elements 1 and 2 as described in this SAP, and as a result of the sediment study (Integral and Anchor QEA 2010) are found to be insufficient to interpret the groundwater chemistry data. For example, the vertical distribution of COPCs in soils, as well as the geologic structure underlying the impoundments, can be evaluated using lithography, grain size, and chemistry data for subsurface soils.

1.9 Special Training and Certification

A technical team will be assembled with the requisite experience and technical skills to successfully complete the soil sampling and analysis. All technical team personnel involved in sample collection will have extensive environmental sampling experience.

Sampling personnel who enter the exclusion zone and contaminant reduction zone (see Attachment A1, Sections 5.1.1 and 5.1.2 for definition and discussion of these zones) may be required to have completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) standard training course and 8-hour refresher courses (see overall HASP [Anchor QEA 2009] for further explanation). The training provides employees with knowledge and skills that enable them to perform their jobs safely and with minimum risk to their personal health. Documentation of course completion will be maintained in personnel files.

Selected laboratories will hold certification through the National Environmental Laboratory Accreditation Program for the methods which that laboratory will perform, where applicable. Training and certification requirements for laboratory personnel will be provided in the laboratory QA plans (to be submitted under separate cover).

1.10 Documents and Records

Records will be maintained documenting all activities and data related to sample collection and to laboratory analyses. Results of data verification and validation activities will also be documented. Procedures for documentation of these activities are described in this section.

The QAPP, FSP (Appendix A), and the HASP Addendum 3 (Attachment A1) for this soil study will be provided to every task participant listed in Section 1.1. Any revisions or amendments to any of the documents that make up the FSP will also be provided to these individuals.

The FSP for the Groundwater SAP (Anchor QEA 2010c) will describe the methods and documentation procedures for collection of soils during establishment of groundwater wells.

1.10.1 Field Records

Components of field documentation relevant to Study Elements 1 and 2 are discussed in Section 3 of the FSP (for soils to be collected in support of Study Element 3, Anchor QEA's field lead will ensure that the field team receives the final, approved version of this QAPP and the Groundwater SAP, including the FSP and soil Job Safety Analyses [Attachment A1], prior to the initiation of field activities). Field records that will be maintained include the following:

- Field logbooks
- Photo documentation
- Field data and sample collection information forms
- Field change request forms (as needed)
- Sample tracking/chain-of-custody (COC) forms.

Observations recorded in the field logbook will be used to provide context and aid in presentation and interpretation of analytical results. Additional details regarding the content and use of these documents are described in Section 3.1 of the FSP. For soils to be collected in support of Study Element 3, documentation is also discussed in the Groundwater FSP.

1.10.2 Laboratory Data Reports

All activities and results related to sample analysis will be documented at each laboratory. Internal laboratory documentation procedures are described in the laboratory QA manuals (to be submitted under separate cover).

Each laboratory will provide a data package for each sample delivery group or analysis batch that is comparable in content to a full Contract Laboratory Program (CLP) package. The format of the data may differ from CLP requirements. Each data package will contain all information required for a complete QA review, including the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this QAPP
- COCs and cooler receipt forms
- A summary of analyte concentrations (to two significant figures, unless otherwise justified), method reporting limits (MRLs), and method detection limits (MDLs) or estimated detection limits (EDLs)
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation, digestion, extraction, dilution, and cleanup logs
- Instrument tuning data
- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and calibration blanks
- Results for all QA/QC checks, including, but not limited to, labeled compounds, surrogate spikes, internal standards, serial dilutions, laboratory control samples, matrix spike samples, matrix spike duplicate samples, and laboratory duplicate samples provided on summary forms
- Instrument data quantification reports for all analyses and samples
- Copies of all laboratory worksheets and standards preparation logs.

Data will be delivered by the laboratories in both hard copy and electronic format to the task QA coordinator, who will be responsible for overseeing data verification and validation and for archiving the final data and data quality reports in the project file. Electronic data deliverables (EDDs) will be compatible with the project database.

1.10.3 Data Quality Documentation

Data verification (i.e., confirming the accuracy and completeness of field and laboratory data) will be completed by the SJRWP technical team for data generated in the field, and by each laboratory for the data that it generates. Data validation reports for chemical analyses will be prepared as described in Section 4 and provided to the task QA coordinator. All changes to data stored in the database will be recorded in the database change log. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratory and during data validation.

1.10.4 Reports and Deliverables

The laboratories will keep the Laboratory QA Coordinator informed of their progress on a weekly basis. The laboratories will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of out-of-control laboratory QC data and any corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

Once all field programs for the Site are complete, a draft Preliminary Site Characterization Report (PSCR) will be prepared and submitted to USEPA, according to the schedule provided in Section 8 of the RI/FS Work Plan (Anchor QEA and Integral 2010b). The draft PSCR will contain sample location maps, and validated analytical chemistry results. Consistent with the 2009 UAO, the draft PSCR will be submitted to USEPA after the completion of all laboratory and data validation work for all of the field studies that will be required for the RI/FS, and according to the schedule provided in Section 8 of the RI/FS Work Plan (Anchor QEA and Integral 2010b). Prior to submittal of the draft PSCR, data will be made available

online according to the schedule for each sampling program provided the RI/FS Work Plan schedule (Anchor QEA and Integral 2010b). Interpretation of the data will be presented in the RI report.

2 DATA GENERATION AND ACQUISITION

This section provides a brief description of the sampling design and outlines the procedures for collecting soil samples. Details of soil sampling methods are provided in the FSP (Appendix A).

2.1 Sampling Design

The sampling design for soil (Table 6) can be summarized as follows:

Soil Collection Areas

Soil collection areas (Figure 3) include:

- Area 1. The unvegetated portion of the upland area west of the impoundments and the area adjacent to the road that provides access into and out of the upland area.
- Area 2. The area underneath I-10. This area has been sampled for the TCRA (Anchor QEA 2010a), and no samples in addition to that effort are planned, but data will be used for Study Elements 1 and 2.
- Area 3. The area of the Site in which the impoundments north of I-10 occur.
    - Groundwater well locations (Figure 3), as described by the Groundwater SAP (Anchor QEA 2010c).

- Area 4. The area in which waste handling south of I-10 may have affected soil, addressed in Soil SAP Addendum 1.
- Background (Figure 6). One or more areas that are near the Site, and near heavily trafficked roadways.

Soil Depth Intervals to be Sampled (Study Elements 1 and 2)

In Area 1 soil samples will be collected at three depth intervals, as follows:

- **Surface soil samples** will be collected from 0 to 6 inches (0 to 15 cm).
- **Shallow subsurface samples** will be collected from 6 to 12 inches (15 to 30 cm).
- **Deep subsurface soil samples** will be collected from 12 to 24 inches (30 to 60 cm).

In Area 3, samples will be collected at 0–6 inch and 6–12 inch depth intervals.

Characterization of the top 1 foot (0–30 cm) of soils at each station will be possible by calculating a depth-weighted concentration using the concentrations in each of the two individual surface intervals, weighted by the percent of the total depth represented by the interval depth.

In background areas, the surface 0–6 inches (0–15 cm) and shallow subsurface 6–12 inches (15–30 cm) intervals will be collected.

For all soil samples collected for Study Elements 1 and 2, an archive sample will be collected at each depth interval and archived for possible future analysis of secondary COPCs.

Analyses of samples collected in the area south of I-10 (Area 4) is discussed in Soil SAP Addendum 1.

Soil Depth Intervals to be Sampled (Study Element 3)

At the location of groundwater wells, soil samples will be collected at 5-foot intervals during well boring. Soils will be composited across the full depth of the interval. Visual observations of lithology will be recorded, and composite samples for all intervals will be analyzed for grain size. Composite samples for the each 5-foot interval will be archived for possible future chemical analyses.

Sample Stations

The following lists the total numbers of location to be collected in each soil collection area, including those collected for the TCRA:

- Area 1: Thirty-one stations
- Area 2: Twelve stations
- Area 3. Six stations at the locations of the borings within the impoundments north of I-10 for geotechnical parameters (SJGB001, SJGB006, SJGB009, SJGB010, SJGB011, SJGB012), and three stations at the locations of groundwater well pairs.
- Area 4. Sample locations described in Soil SAP Addendum 1.

Locations of all of these stations (except those in Area 4) are shown in Figure 3.

Specific locations of background stations are not defined, but 20 background locations will be sampled from one or more of the public areas shown in Figure 6. Specific locations will be determined on the basis of safety, accessibility, and permissions.

Samples to be collected from groundwater well boreholes will be collected in the groundwater well locations (Figure 3).

Conditional Samples

- Chemical analyses of soils collected from each 5-foot interval of groundwater well borings will be analyzed if soil and sediment chemical data for the nature and extent and exposure objectives are not sufficient for interpretation of groundwater chemistry data.

LiDAR Data

LiDAR data for the Site will be obtained from the Houston-Galveston Area Council. It will be interpreted using various geographic information system software applications, and will be used to define surface hydrology flow paths on the upland area west of the impoundments. No field activities will be performed to collect or evaluate these data.

2.2 Sampling Methods

Sampling methods that will be used to collect the soil samples are presented in the following section. Sampling methods are described in detail in the FSP (Appendix A). Any sampling methods that may be required for collecting soil south of I-10 that differ from methods described below and in the FSP are described in Soil SAP Addendum 1.

All surface and shallow subsurface soil samples for characterization of nature and extent, for exposure of ecological receptors, and for characterization of human health exposure will be collected from 0 to 6 inches (0 to 15 cm) and from 6 to 12 inches (15 to 30 cm); deep subsurface soil samples will be collected from 12 to 24 inches (30 to 60 cm) at selected locations (Figure 3).

Depending upon the conditions encountered in the field, sampling equipment may include stainless-steel shovels, trowels, and spoons. One sample at each of the surface soil and subsurface soil intervals will be collected at each sampling location. The soil samples collected at each station will be placed into a decontaminated stainless-steel bowl and homogenized using a stainless-steel spoon until the soil attains a visually uniform color and texture. Soils will then be removed from the bowl for the laboratory analyses or for archiving.

Further details of the soil sampling methods, collection, and sample processing can be found in the FSP. Locations of surface soil sampling stations are shown in Figure 3; locations in background areas are shown in Figure 6.

2.3 Sample Handling and Custody

Principal documents used to identify samples and to document sample possession will be field logbooks and COC records. Custody will be documented for all samples at all stages of the analytical or transfer process. COC procedures for sample handling prior to delivery to the laboratory are outlined in Section 3.3 of the FSP.

Upon receipt of samples at each laboratory, the physical integrity of the containers and seals will be checked, and the samples will be inventoried by comparing sample labels to those on the COC forms. The laboratory will include the COC and shipping container receipt forms in the data package. Any breaks in the COC or non-conformance will be noted and reported in writing to the project laboratory coordinator within 24 hours of receipt of the samples. The laboratory QA plan (provided under separate cover) includes procedures used for accepting custody of samples and documenting samples at the laboratory. The laboratory project manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratory.

All samples will be stored in accordance with Table 7. Samples for chemical analyses will be stored under refrigeration (4 ± 2°C). Aliquots of the samples submitted to the analytical laboratory for long-term archiving for future analysis will be stored at –20°C. Each

laboratory will maintain COC documentation and documentation of proper storage conditions for the entire time that the samples are in its possession.

The laboratories will not dispose of the samples for this task until authorized to do so by the task QA coordinator. After authorization is obtained, each laboratory will dispose of samples, as appropriate, based on matrix, analytical results, and information received from the client.

2.4 Laboratory and Analytical Methods

Soil samples collected for this study will be analyzed for ancillary parameters (TOC, grain size, and percent moisture) and primary COPCs (metals, dioxin/furans, bis(2-ethylhexyl)phthalate), and may be analyzed for secondary COPCs (polychlorinated biphenyl [PCB] congeners, and semivolatile organic compounds [SVOCs]) (Table 5). The proposed laboratory methods are described below and are summarized in Table 8.

TOC in soil will be analyzed by USEPA Method 9060A. Samples will be pretreated with hydrochloric acid to remove inorganic carbon, dried at 70°C, and analyzed by combustion in an induction furnace (USEPA 2009b).

Grain size distribution will be determined according to ASTM Methods D-422 and D-1140 (ASTM 2009), with modifications described in USEPA (1986). Organic material in the samples will not be oxidized prior to analysis.

Percent moisture will be determined according to USEPA Method 160.3 (USEPA 1971). These results will be used to calculate analyte concentrations on a dry-weight basis and will also be reported in the database.

Dioxins and furans in soil samples will be extracted and analyzed in accordance with USEPA Method 1613B (USEPA 1994). All extracts will undergo silica gel cleanup. Additional cleanup procedures will be used as necessary. Samples will be analyzed by high-resolution gas chromatography with high-resolution mass spectrometry (HRGC/HRMS). Detection

limits are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer.

Soils analyzed for metals other than mercury will be digested with strong acid per USEPA Method 3050 and analyzed by inductively coupled plasma-atomic emission spectrometry per USEPA Method 6010B, or by inductively coupled plasma/mass spectrometry per USEPA Method 6020 (USEPA 2009b).

If secondary COPCs are analyzed in soil, the following methods will be used. PCB congeners will be extracted and analyzed in accordance with USEPA Method 1668A (USEPA 1999a). All extracts will undergo silica gel cleanup. Additional cleanup procedures will be used as necessary. Samples will be analyzed by HRGC/HRMS. Detection limits are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer.

SVOCs will be extracted using Soxhlet or pressurized fluid extraction procedures, processed through gel permeation chromatography, and analyzed by gas chromatography/mass spectrometry in accordance with USEPA Method 8270C (USEPA 2009b). Tentatively identified compounds will not be reported. For analysis of soil, sample modifications such as use of selected ion monitoring or large volume injectors may be made to these methods to improve MRLs.

2.5 Quality Control

QC samples will be prepared in the field and at each laboratory to monitor the bias and precision of the sample collection and analysis procedures.

2.5.1 Field Quality Control

Field QC samples for this study will include field split samples (homogenization duplicate), equipment filter wipes, filter blanks, and Standard Reference Material (SRM).

Field split samples will be collected at an approximate frequency of one for every 20 field samples processed. Equipment filter wipes will consist of clean, ashless filter papers supplied

by the analytical laboratory. Equipment filter wipes will be collected at a frequency of one for every 20 field samples processed for each type of nondedicated equipment in direct contact with the soils being collected. One filter blank will be collected for each lot of filter wipes used during the field effort. One SRM for soils will be submitted from the field and analyzed for dioxins and furans.

Procedures for preparing field split samples, equipment wipes, and SRMs are presented in Section 2.2 of the FSP. Validation criteria and procedures for field QC samples are described in Sections 4.1 and 4.2 of this SAP.

2.5.2 Laboratory Quality Control

Extensive and detailed requirements for laboratory QC procedures are provided in the methods that will be used for this investigation (Table 8). QC requirements include control limits and requirements for corrective action in many cases. QC procedures will be completed by each laboratory, as required by each protocol and as indicated in this QAPP. Laboratory QC procedures are addressed for chemical and physical laboratories below.

The overall quality objective for this task is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. The QA procedures and measurements that will be used for this project are based on USEPA guidance (USEPA 1971, 1986, 1994, 1999a, 2009b) and on established laboratory methods from other sources (ASTM 2009).

2.5.2.1 Chemistry Laboratory QA

The frequency of analysis for laboratory control samples and method blanks will be one for every 20 samples or one per extraction batch, whichever is more frequent. Labeled compounds and internal standards will be added to every field sample and QC sample, as required. Calibration procedures will be completed at the frequency specified in the method description. Performance-based control limits have been established by the laboratory. These and all other control limits specified in the method descriptions will be used by the laboratory to establish the acceptability of the data or the need for reanalysis of the samples. Laboratory control limits for recoveries of labeled compounds and laboratory control

samples, and for relative percent difference (RPD) of matrix spike duplicates and laboratory duplicates, are provided in each laboratory's QA manual (Appendix B).

PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are commonly used to assess the quality of environmental data. Bias represents the degree to which a measured concentration conforms to the reference value. The results for matrix spikes, laboratory control samples, field blanks, and method blanks will be reviewed to evaluate bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = [(M-U) / C] \times 100 \quad (1\text{-}1)$$

where:

%R	=	percent recovery
M	=	measured concentration in the spiked sample
U	=	measured concentration in the unspiked sample
C	=	concentration of the added spike

The following calculation is used to determine percent recovery for a laboratory control sample or reference material:

$$\%R = (M / C) \times 100 \quad (1\text{-}2)$$

where:

%R	=	percent recovery
M	=	measured concentration in the spiked sample
U	=	measured concentration in the unspiked sample
C	=	concentration of the added spike

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analytes detected in the field or in method blanks will be evaluated as potential indicators of bias.

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of matrix spike duplicates, laboratory duplicates, field splits, and field replicates. Precision is expressed in terms of the relative standard deviation for three or more measurements and the RPD for two measurements. The following equation is used to calculate the RPD between measurements:

$$RPD = |[(C1-C2) / ((C1 + C2) / 2)]| \times 100 \quad (1\text{-}3)$$

where:

RPD = relative percent difference
C1 = first measurement
C2 = second measurement

The relative standard deviation is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

Completeness will be calculated as the ratio of usable data (i.e., unqualified data and *U*- or *J*-qualified data) to generated data, expressed as a percentage. Completeness will be calculated for each suite of analytes for each sample type and sampling event.

Additional laboratory QC results will be evaluated to provide supplementary information regarding overall quality of the data, performance of instruments and measurement systems, and sample-specific matrix effects.

QC samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table 8. All QC requirements will be completed by each laboratory as described in the protocols, including the following (as applicable to each analysis):

- Instrument tuning
- Initial calibration
- Initial calibration verification

- Continuing calibration verification
- Calibration or instrument blanks
- Method blanks
- Laboratory control samples
- Internal standards
- Surrogate spikes/labeled compounds
- Matrix spikes
- Matrix spike duplicates or laboratory duplicates

To alert the data user to possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated QC samples or procedures do not meet control limits. Laboratory control limits for the methods that will be used for this Site investigation are provided in Table 9 and in the laboratory QA manuals (to be provided under separate cover). Data validation criteria and procedures are described in Section 4.

MRLs reflect the sensitivity of the analysis. Target MRLs for this study are summarized in Table 9 where possible.

Method detection limits (MDLs) will be determined by each laboratory for each analyte, as required by USEPA (2009b). MDLs are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix (e.g., sand or distilled water) with 99 percent confidence that a false positive result has not been reported. MRLs are established by the laboratories at levels above the MDLs for the project analytes. The MRL values are based on the laboratories' experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system in environmental samples. For this task, the concentration of the lowest standard in the initial calibration curve for each analysis is at the level of the MRL. This allows reliable quantification of concentrations to the MRL in the absence of matrix interferences.

Analyte concentrations for this task will be reported to the sample-specific MDLs as described in their respective analytical methods, listed in Table 8. Analytes detected at concentrations between the MRL and the MDL will be reported with a *J* qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range).

Non-detects will be reported at the MDL for all analytes. The MRLs and MDLs will be adjusted by each laboratory, as necessary, to reflect sample dilution, percent moisture, and/or matrix interference.

2.5.2.2 Representativeness and Comparability of All Data

Representativeness and comparability are qualitative QA/QC parameters. Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

Comparability is the qualitative similarity of one dataset to another (i.e., the extent to which different datasets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by USEPA and are commonly used for soil studies.

2.6 Instrument and Equipment Testing, Inspection, and Maintenance

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the laboratory consistent with the requirements identified in the laboratory's SOPs and manufacturer instructions. In addition, each of the specified analytical methods provides protocols for proper instrument setup and tuning, and critical operating parameters. Instrument maintenance and repair will be documented in the maintenance log or record book.

2.7 Inspection and Acceptance of Supplies and Consumables

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and QC purposes.

During sample collection, the quality of laboratory water used for decontamination will be documented at the laboratory that provides that water. Precleaned sample jars (with documentation) will be provided by the laboratories. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in the laboratory SOPs and QA manuals. All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (i.e., for supplies used in the field) or the laboratories.

2.8 Non-Direct Measurements

Existing chemical data from previous 2010 investigations performed for the RI/FS will be used for this study.

2.9 Data Management

During field, laboratory, and data evaluation operations, effective data management is critical to providing consistent, accurate, and defensible data and data products. Data management systems and procedures will be used to establish and maintain an efficient organization of the environmental information collected. Procedures and standards for conducting specific data management tasks (i.e., creation, acquisition, handling, storage, and distribution of data) are documented in the RI/FS data management manual. Essential elements of data management and reporting activities associated with soil sampling are discussed in the following sections.

Project data will be maintained in a relational database designed to accommodate all the types of environmental measurements that will be made during the RI/FS for the Site, as described in the data management plan, which is included as Appendix B of the RI/FS Work Plan (Anchor QEA and Integral 2010b). On-line access to the database will be provided to members of the project team and regulatory oversight bodies through a browser-based interface.

2.9.1 Field Data

Daily field records (a combination of field logbooks, field forms, global positioning system [GPS] records, and COC forms) will make up the main documentation for field activities. Detailed guidelines for entry of information during field sampling are provided in the FSP, which is included as Appendix A to this SAP. Upon completion of sampling, hardcopy notes and forms will be scanned to create an electronic record for use in creating the draft PSCR. Information on sampling locations, dates, depths, equipment, and other conditions and sample identifiers, will be entered into the project database. One hundred percent of hand-entered data will be verified based on hard copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

2.9.2 Laboratory Data

The analytical laboratories will each submit data in both electronic and hard-copy format. The project database administrator or his designated data manager will specify the appropriate format for EDDs to the laboratory, and the project data manager and laboratory coordinator will discuss these specifications with laboratory QA managers prior to data delivery and tailor them as necessary to specific laboratory capabilities. QA checks of format and consistency will be applied to EDDs received from the laboratory. After any issues have been resolved, the data will be loaded into the RI/FS project database. Each dataset loaded will be linked to the electronic document of the relevant laboratory data package. Data summaries will be produced from the database for use by data validators. Validators will return edited versions of these summaries, and the edits will then be incorporated into the database. An automated change log will be maintained by the database so that the history of all such edits is maintained, and the provenance of each data value can be determined.

3 ASSESSMENT AND OVERSIGHT

This task will rely on the knowledge and expertise of the SJRWP technical team, as described in the RI/FS Work Plan (Anchor QEA and Integral 2010). The field teams and laboratory will stay in verbal contact with the project managers and QA coordinator throughout this task. This level of communication will serve to keep the management team informed about activities and events, and will allow for informal but continuous task oversight.

3.1 Assessment and Response Actions

Assessment activities will include readiness reviews by the field lead prior to sampling, by the database administrator prior to release of the final data to the data users, and internal review while work is in progress. An informal technical systems audit may be conducted if problems are encountered during any phase of this project.

The first readiness review will be conducted by the field lead prior to field sampling to verify that all field equipment is ready for transfer to the Site. The field lead will also verify that the field team and any subcontractors have been scheduled and briefed and that the contracts for the subcontractors have been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed by the database administrator before final data are released for use to verify that all results have been received from each laboratory, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the database administrator, the task QA coordinator, or their designee. Data will not be released for final use until all data have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the SJRWP project coordinator and data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this task will be completed throughout the course of all sampling, laboratory, data validation, data management, and data interpretation activities to ensure that every phase of work is accurate

and complete and follows the QA procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the SJRWP technical team coordinator and SJRWP project coordinator.

The laboratory will be required to have implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Details are provided in the laboratory QA plan (to be submitted under separate cover).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. If completed, these audits will be conducted by the task QA coordinator or designee, or by the laboratory, as appropriate. These audits may consist of on-site reviews of any phase of field or laboratory activities or data management. Results of any audits will be provided in the draft PSCR.

Any task team member who discovers or suspects a nonconformance is responsible for reporting the nonconformance to the task manager, the task QA coordinator, or the laboratory project or QA manager, as applicable. The task QA coordinator will ensure that no additional work dependent on the nonconforming activity is performed until a confirmed nonconformance is corrected. Any confirmed nonconformance issues will be relayed to the SJRWP technical team coordinator.

3.2 Reports to Management

The laboratory will keep the laboratory coordinator informed of their progress on a weekly basis. The laboratory will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of any laboratory QC data outside of control limits and any corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

The task QA coordinator will provide this information to the Integral project manager.

Individual laboratories will be required to have implemented routine systems of reporting nonconformance issues and their resolution. These procedures are described in the laboratory QA manual (Appendix B). Laboratory nonconformance issues will also be described in the draft PSCR if they affect the quality of the data.

Data packages and EDDs will be prepared by each laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances (if applicable), and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Data validation reports will be prepared following receipt of the complete laboratory data packages for each sample delivery group. These reports will be provided to the task QA coordinator when validation is completed for each parameter. A summary of any significant data quality issues will be provided to USEPA with the data report.

4 DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the data report.

4.1 Criteria for Data Review, Verification, and Validation

Field and laboratory data for this task will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

Data verification and validation for dioxins and furans, metals, and organic compounds will be completed in accordance with Guidance on Environmental Data Verification and Validation (USEPA 2002a) and according to methods described in USEPA's National Functional Guidelines for inorganic and organic data review (USEPA 1999b, 2002b, 2005b). Performance-based control limits established by the laboratories and control limits provided in the method protocols will be used to evaluate data quality and determine the need for data qualification. Performance-based control limits are established periodically by the laboratory. Current values will be provided in the laboratory QA plans (to be submitted under separate cover), as applicable.

Results for field splits will be evaluated against a control limit of 50% relative percent difference (RPD). Data will not be qualified as estimated if this control limit is exceeded, but RPD results will be tabulated, and any exceedances will be discussed in the draft PSCR. Equipment wipe blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in the functional guidelines for data review (USEPA 1999b, 2002a,b, 2005).

Data will be rejected if control limits for acceptance of data are not met, as described in USEPA (USEPA 1999b, 2002a,b, 2005).

4.2 Verification and Validation Methods

Both the chemical and conventional analyses will undergo verification and validation, as described below.

Field data will be verified during preparation of samples and COC forms. Field data and COC forms will be reviewed daily by the field lead. After field data are entered into the project database, 100 percent verification of the entries will be completed by a second party to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is released for use.

Data verification and validation will be completed as described in Section 4.1 by either Integral or a data validation firm. The first data package generated will be fully validated, equivalent to a Stage 4 validation as described in USEPA (2009a). If no major problems are encountered during validation of this package, full validation will be completed at a rate of approximately 30 percent of the dioxin and furan samples. Validation for the remaining data will be based on a review of the sample and QC data, equivalent to a Stage 2B validation. If problems are encountered, the laboratory will be contacted for resolution. Additional full validation will be completed if required to fully assess the quality of the data to verify that the laboratory errors have been addressed.

The accuracy and completion of the database will be verified at the laboratory when the EDDs are prepared and again as part of data validation. Ten percent of entries to the database from laboratory EDDs will be checked against hard-copy data packages. In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use.

Reporting limits for non-detects will be compared to the MRL goals to evaluate method sensitivity for each sample. Any exceedance of actual MRLs over the target MRLs will be discussed in the letter report.

4.3 Reconciliation with User Requirements

Both the chemical and conventional analyses will undergo reconciliation with user requirements, as described below.

The goal of data validation is to determine the quality of each data result and to identify those that do not meet the task measurement quality objectives. Nonconforming data may be qualified as estimated (i.e., a *J* qualifier will be applied to the result) or rejected as unusable (i.e., an *R* qualifier will be applied to the result) during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the draft PSCR.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. However, these data are less precise or less accurate than unqualified data. Data users, in cooperation with the SJRWP technical team coordinator and the task QA coordinator, are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses.

5 REFERENCES

Anchor QEA, 2009. Health and Safety Plan San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS.

Anchor QEA, 2010a. TXDOT Right-of-Way Field Sampling Plan. Prepared for McGinnes Industrial Maintenance Corporation and International Paper Company. Anchor QEA, Ocean Springs, MS.

Anchor QEA, 2010b. Draft TxDOT Right-of-Way Data Report. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS.

Anchor QEA, 2010c. Draft Groundwater Study Sampling and Analysis Plan, San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS.

Anchor QEA and Integral, 2010a. Draft Addendum to the Removal Action Work Plan (RAWP) for the Time Critical Removal Action (TCRA), San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS, and Integral Consulting, Seattle, WA.

Anchor QEA and Integral, 2010b. Revised Remedial Investigation/Feasibility Study Work Plan San Jacinto Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS, and Integral Consulting, Seattle, WA.

ASTM, 2009. Annual Book of ASTM Standards. Volume 04.08. American Society for Testing and Materials, Philadelphia, PA.

Boerngen, J.G., and H.T. Shacklette, 1981. Chemical Analyses of Soils and Other Surficial Materials of the Conterminous United States: U.S. Geological Survey Open-File

Report 81-197, U.S. Geological Survey, Denver, CO. Available online at http://tin.er.usgs.gov/ussoils/

Gonzales, A.G., 2007. Use and Misuse of Supervised Pattern Recognition Methods for Interpreting Compositional Data. *J. Chromatogr.* A 1158:215-225.

Integral and Anchor QEA, 2010. Sampling and Analysis Plan: Sediment Study San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS, and Integral Consulting Inc., Seattle, WA.

Johnson, G., R. Ehrlich, W. Full, and S. Ramos, 2007. Principal Component Analysis and Receptor Models in Environmental Forensics. pp. 207–261. In: *Introduction to Environmental Forensics*. Second Edition. B. Murphy and R.D. Morrison (eds.). Academic Press.

Myers, J.C., 1997. Geostatistical Error Management: Quantifying Uncertainty for Environmental Sampling and Mapping. Van Nostrand Reinhold Publishing, New York, NY.

TCEQ, 2003. Guidance for Assessing Texas Surface and Finished Drinking Water Quality Data, 2004. Texas Commission on Environmental Quality, Office of Compliance and Enforcement, Monitoring Operations Division, Surface Water Quality Monitoring Program.

TCEQ. 2010. TRRP Protective Concentration Levels. Available at: http://www.tceq.state.tx.us/remediation/trrp/trrppcls.html. Accessed December 1, 2010. Texas Commission on Environmental Quality.

TCEQ and USEPA, 2006. Screening Site Assessment Report San Jacinto River Waste Pits, Channelview, Harris County, Texas. TXN000606611. Texas Commission on Environmental Quality and U.S. Environmental Protection Agency.

TSDH, 1966. Investigation of Industrial Waste Disposal – Champion Paper, Inc. Pasadena. Texas State Department of Health Memorandum from Stanley W. Thompson, P.E., Regional Engineer, to the Director of the Division of Water Pollution Control. May 6, 1966.

Tzhone, S., 2010. Personal communication (e-mail to D. Keith, Anchor QEA, Ocean Springs, MS, and J. Sampson, Integral Consulting, Seattle, WA, dated May 28, 2010, regarding results of San Jacinto River Waste Pits VOC analyses). U.S. Environmental Protection Agency, Region 6, Dallas, TX.

University of Houston and Parsons, 2006. Total Maximum Daily Loads for Dioxins in the Houston Ship Channel. Contract No. 582-6-70860, Work Order No. 582-6-70860-02. Quarterly report No. 3. Prepared in cooperation with the Texas Commission on Environmental Quality and the U.S. Environmental Protection Agency. University of Houston and Parsons Water & Infrastructure. Available at: http://www.tceq.state.tx.us/assets/public/implementation/water/tmdl/26hscdioxin/26-all-data-compiled-q3-fy06.pdf.

USEPA, 1971. Method 160.3: Total Residue by Drying Oven. Methods for the Chemical Analysis of Water and Wastes. EPA/600/4-79-020. U.S. Environmental Protection Agency, Office of Water, Washington, DC.

USEPA, 1986. Puget Sound Estuary Program: Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound. Final Report. Prepared for U.S. EPA, Region 10, Office of Puget Sound, Seattle, WA and the U.S. Army Corps of Engineers, Seattle District, Seattle, WA. Tetra Tech, Inc., Bellevue, WA.

USEPA, 1994. Method 1613, Tetra- Through Octa-chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Washington, DC.

USEPA, 1999a. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. EPA-821-R-00-002. December 1999. U.S. Environmental Protection Agency, Office of Water, Washington, DC.

USEPA, 1999b. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA 540/R-99/008. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 2000. Exposure and Human Health Reassessment of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) and Related Compounds. EPA/600/P-00/001Bc. Draft Final Report. U.S. Environmental Protection Agency, National Center for Environmental Assessment, Washington, DC.

USEPA, 2001a. EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. EPA/240/B-01/003. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA, 2001b. Denver Front Range Study. Dioxins in Surface Soil. Study 1: Characterization of Dioxins, Furans and PCBs In Soil Samples Collected from the Denver Front Range Area. U.S. Environmental Protection Agency, Region 8, with technical assistance from Syracuse Research Corporation, Inc. and Gannett Fleming, Inc. July, 2001.

USEPA, 2002a. Guidance on Environmental Data Verification and Validation. EPA QA/G-8. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA, 2002b. Guidance for Quality Assurance Project Plans. EPA QA/G-5. EPA/240/R-02/009. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA, 2002c. Guidance for Comparing Background and Chemical Concentrations in Soil for CERCLA Sites. EPA 540-R-01-003. OSWER 9285.7-41. September. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 2004. USEPA. Dioxin Reassessment. National Academy of Sciences (NAS) Review Draft. EPA/600/P-00/001Cb. U.S. Environmental Protection Agency, Washington, DC.

USEPA 2005a. Guidance for Developing Ecological Soil Screening Levels. OSWER Directive 9285.7-55. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, DC.

USEPA, 2005b. USEPA Contract Laboratory Program Functional Guidelines for Chlorinated Dioxin/Furan Data Review. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. EPA/240/B-06/001. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA, 2008. Framework for Investigating Asbestos-Contaminated Superfund Sites. OSWER Directive #9200.0-68. U.S. Environmental Protection Agency, Asbestos Committee of the Technical Review Workgroup of the Office of Solid Waste and Emergency Response, Washington, DC.

USEPA, 2009a. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 2009b. SW-846 On-line, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods. U.S. Environmental Protection Agency. http://www.epa.gov/epaoswer/hazwaste/test/main.htm. Last updated March 23, 2009.

USEPA, 2009c. Unilateral Administrative Order for Remedial Investigation/Feasibility Study. U.S. EPA Region 6 CERCLA Docket No. 06-03-10. In the matter of: San Jacinto River Waste Pits Superfund Site Pasadena, Texas. International Paper Company, Inc. & McGinnes Industrial Management Corporation, respondents.

USEPA, 2009d. Draft Recommended Interim Preliminary Remediation Goals for Dioxin in Soil at CERCLA and RCRA Sites. OSWER 9200.3-56. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation, Washington, DC.

USEPA, 2010a. Request for a Time Critical Removal Action at the San Jacinto River Waste Pits Site, Harris County, Texas. Internal memorandum from V. Leos, Remedial Project Manager Remedial Branch, to S. Coleman, P.E. Director Superfund Division, dated April 2, 2010. U.S. Environmental Protection Agency, Region 6, Dallas, TX.

USEPA. 2010b. Regional Screening Levels. United States Environmental Protection Agency. Last updated May 2010. Available at: http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/Generic_Tables/index.htm

van den Berg, M., L.S. Birnbaum, M. Denison, M. De Vito, W. Farland, M. Freeley, H. Fiedler, H. Hakansson, A. Handberg, L. Haws, M. Rose, S. Safe, D. Schrenk, C. Tohyama, A. Tritscher, J. Tuomisto, M. Tysklind, N. Walker, and R.E. Peterson, 2006. The 2005 World Health Organization reevaluation of human and mammalian toxic equivalency factors for dioxins and dioxin-like compounds. *Tox. Sci.* 2(93):223–241.

TABLES

Table 1

Results of Screening of TCRA Soils Collected From the TxDOT ROW[a]

Analyte	Screening Level for Industrial Soil [b]	Surface Soil			Subsurface Soil			All Soil		
		FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
Metals (mg/kg - dw)										
Aluminum	9.9E+05 nc	11/11	9,070	--	1/1	2,240	--	12/12	9,070	--
Arsenic	1.6E+00 c	11/11	3.9	--	1/1	1.62	--	12/12	3.9	--
Barium	1.9E+05 nc	11/11	255	--	1/1	42.9	--	12/12	255	--
Cadmium	8.0E+02 nc	11/11	0.44 J	--	1/1	0.07 J	--	12/12	0.44 J	--
Chromium	1.5E+06 [c] nc	11/11	61.7	--	1/1	4.9	--	12/12	61.7	--
Cobalt	3.0E+02 nc	11/11	32.2	--	1/1	2.2	--	12/12	32.2	--
Copper	4.1E+04 nc	11/11	39.5	--	1/1	4.5	--	12/12	39.5	--
Lead	8.0E+02 nc	11/11	273	--	1/1	19	--	12/12	273	--
Magnesium	NV	11/11	3,000 J	--	1/1	1,110 J	--	12/12	3,000 J	--
Manganese	2.3E+04 nc	11/11	969	--	1/1	73.7	--	12/12	969	--
Mercury	3.1E+02 nc	11/11	0.081	--	1/1	0.017 J	--	12/12	0.081	--
Nickel	2.0E+04 nc	11/11	11.9	--	1/1	3.18 J	--	12/12	11.9	--
Thallium	7.8E+01 nc	0/11	--	0.5	0/1	--	0.4	0/12	--	0.5
Vanadium	5.2E+03 nc	11/11	33.5	--	1/1	9.1	--	12/12	33.5	--
Zinc	3.1E+05 nc	11/11	188	--	1/1	40.1	--	12/12	188	--
Organics										
Dioxins/Furans (ng/kg=dw)(as TEQ)										
TEQdfMamDL1/2	1.8E+01 [d] c	11/11	66.1 J	--	1/1	1.22 J	--	12/12	66.1 J	--
PCBs (ug/kg-dw)										
Aroclor 1016	2.1E+04 c	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1221	5.4E+02 c	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1232	5.4E+02 c	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1242	7.4E+02 c	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1248	7.4E+02 c	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1254	7.4E+02 c	1/11	130	19	1/1	46 J	--	2/12	130	19
Aroclor 1260	7.4E+02 c	3/11	44 J	19	0/1	--	19	3/12	44 J	19
Aroclor 1262	NV	0/11	--	19	0/1	--	19	0/12	--	19
Aroclor 1268	NV	0/11	--	19	0/1	--	19	0/12	--	19
PCB 77	1.1E+02 c	11/11	0.13 J	--	0/1	--	0.0035	11/12	0.13 J	0.0035
PCB 81	3.8E+01 c	3/11	0.0041 J	0.0023	0/1	--	0.0003	3/12	0.0041 J	0.0023
PCB 105	3.8E+02 c	11/11	4.3	--	1/1	0.016 J	--	12/12	4.3	--
PCB 114	3.8E+02 c	8/11	0.25 J	0.0098	0/1	--	0.0013	9/12	0.25 J	0.0098
PCB 118	3.8E+02 c	11/11	10.5	--	1/1	0.0043 J	--	12/12	10.5	--
PCB 123	3.8E+02 c	8/11	0.15 J	0.021	0/1	--	0.0013	9/12	0.15 J	0.021
PCB 126	1.1E-01 c	6/11	0.025 J	0.0098	0/1	--	0.0012	6/12	0.025 J	0.0098
PCB 156	3.8E+02 c									
PCB 157	3.8E+02 c	11/11	1.8 [e]	--	1/1	0.009 [e] J	--	12/12	1.8 [e]	--
PCB 167	3.8E+02 c	11/11	0.52 J	--	0/1		0.0029	11/12	0.52 J	0.0029
PCB 169	3.8E-01 c	3/11	0.0095 J	0.0026	0/1	--	0.0008	3/12	0.0095 J	0.0026
PCB 189	3.8E+02 c	10/11	0.11 J	0.0071	0/1		0.0007	10/12	0.11 J	0.0071
Total PCBs (as Total Aroclors)	7.4E+02 c	Not calculated, concentration is not meaningful given high non-detection status for individual Aroclors								
Total PCBs (as Total Dioxin-Like Congeners)	7.4E+02 c	11/11	18	--	1/1	0.075	--	12/12	18	--

Table 1

Results of Screening of TCRA Soils Collected From the TxDOT ROW[a]

Analyte	Screening Level for Industrial Soil [b]		Surface Soil			Subsurface Soil			All Soil		
			FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
Pesticides (ug/kg-dw)											
Carbazole	9.5E+05	c	6/11	210 J	12	1/1	59 J	--	7/12	210 J	12
SVOCs (ug/kg-dw)											
1,2,4-Trichlorobenzene	9.9E+04	c	0/11	--	0.34	0/1	--	0.26	0/12	--	0.34
1,2-Dichlorobenzene	9.8E+06	nc	0/11	--	0.34	0/1	--	0.26	0/12	--	0.34
1,3-Dichlorobenzene	8.8E+04	nc	0/11	--	0.36	0/1	--	0.30	0/12	--	0.36
1,4-Dichlorobenzene	1.2E+04	c	0/11	--	0.37	0/1	--	0.31	0/12	--	0.37
2,4,6-Trichlorophenol	1.6E+05	c	0/11	--	16	0/1	--	15	0/12	--	16
2,4-Dichlorophenol	1.8E+06	nc	0/11	--	19	0/1	--	17	0/12	--	19
Acenaphthene	3.3E+07	nc	3/11	220 J	15	1/1	250 J	--	4/12	250 J	15
bis(2-Ethylhexyl)phthalate	1.2E+05	c	11/11	140 J	--	1/1	100 J	--	12/12	140 J	--
Fluorene	2.2E+07	nc	3/11	120 J	15	1/1	130 J	--	4/12	130 J	15
Hexachlorobenzene	1.1E+03	c	0/11	--	17	0/1	--	15	0/12	--	17
Naphthalene	1.8E+04	c	1/11	110 J	16	0/1	--	15	1/12	110 J	16
Pentachlorophenol	2.7E+03	c	0/11	--	140	0/1	--	130	0/12	--	140
Phenanthrene	1.9E+07	nc	10/11	1,000	10	1/1	770	--	11/12	1,000	10
Phenol	1.8E+08	nc	0/11	--	22	0/1	--	20	0/12	--	22
VOCs (ug/kg-dw)											
1,2,3-Trichlorobenzene	4.9E+05	nc	0/11	--	0.31	0/1	--	0.26	0/12	--	0.31
Chloroform	1.5E+03	c	0/11	--	0.33	0/1	--	0.27	0/12	--	0.33

Notes

-- = Not applicable
c = Screening level is based on carcinogenic endpoint
FOD = frequency of detection
J = Result is less than the method reporting limit but greater than or equal to the method detection limit, and the concentration is an estimated value.
nc = Screening level is based on a noncancer endpoint
NV = No value available
RSL = regional screening level
SVOC = semivolatile organic compound
VOC = volatile organic compound

a - TxDOT 2010 soil from within Site boundary.
b - Screening levels were selected using the following tiered approach:
Tier 1: USEPA 2010b. USEPA RSLs for Industrial Soil- available at: http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/Generic_Tables/index.htm. Accessed Dec 7, 2010.
Tier 2: TCEQ 2010. TCEQ Tier 1 Commercial/Industrial PCLs for 30 acre source area. Available at: http://www.tceq.state.tx.us/remediation/trrp/trrppcls.html
Values for 1,3-dichlorobenzene, carbazole, phenanthrene, and thallium are from TCEQ, whereas the remainder are from USEPA (2010b).
c - Value is for chromium(III). Value for chromium(VI) is lower.
d - Value shown is the cancer risk based RSL for industrial/commercial soils from USEPA (2010b). The value of 950 ng/kg dw proposed as a draft preliminary remediation goal for industrial/commercial soil (USEPA 2009d) should also be considered.
e - Analytes are co-eluted; based on assumption of additive risk do not exceed RSLs.
Shaded cells exceed the screening level.

Table 2
Results of Screening of TCRA Soils Collected from the Upland Sand Separation Area West of the Impoundments

Analyte	Screening Level for Industrial Soil [a]		Surface Soil			Subsurface Soil			All Soil		
			FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
Conventionals (mg/kg-dw)											
Cyanide	2.0E+07		1/3	0.285	0.07	1/3	4.22	0.07	2/6	4.22	0.07
Metals (mg/kg - dw)											
Antimony	4.1E+02	nc	1/3	4.45 J	3.5	1/3	4.45 J	3.4	2/6	4.45 J	3.5
Arsenic	1.6E+00	c	3/3	2.46	--	3/3	2.23	--	6/6	2.46	--
Beryllium	2.0E+03	nc	3/3	0.38 J	--	2/3	0.395 J	0.1	5/6	0.395 J	0.1
Cadmium	8.0E+02	nc	3/3	0.141	--	3/3	0.17	--	6/6	0.17	--
Chromium	1.5E+06 [b]	nc	3/3	8.88	--	3/3	8.21	--	6/6	8.88	--
Copper	4.1E+04	nc	3/3	10.3	--	3/3	7.15	--	6/6	10.3	--
Lead	8.0E+02	nc	2/3	23.4 J	3.5	2/3	65.2	3.3	4/6	65.2	3.5
Mercury	3.4E+01	nc	3/3	0.163	--	3/3	0.034	--	6/6	0.163	--
Nickel	2.0E+04	nc	3/3	6.3	--	3/3	4.9 J	--	6/6	6.3	--
Selenium	5.1E+03	nc	0/3	--	4.8	0/3	--	4.7	0/6	--	4.8
Silver	5.1E+03	nc	0/3	--	0.6	0/3	--	0.7	0/6	--	0.7
Thallium	7.8E+01	nc	1/3	10.5 J	3.5	2/3	5.55 J	3.4	3/6	10.5 J	3.5
Zinc	3.1E+05	nc	3/3	71.7	--	3/3	66.3	--	6/6	72	--
Organics											
Dioxin and Furans (ng/kg-dw)(as TEQ)											
TEQdfMamDL/2	1.8E+1 [c]	c	13/13	27.2 J	--	13/13	26.7 J	--	25/25	27.2 J	--
Organochlorine Pesticides (ug/kg - dw)											
4,4'-DDD	7.2E+03	c	0/3	--	1	0/3	--	1	0/6	--	1
4,4'-DDE	5.1E+03	c	0/3	--	1.6	0/3	--	1.6	0/6	--	1.6
4,4'-DDT	7.0E+03	c	1/3	1 J	0.85	3/3	1.6 J	--	4/6	1.6 J	0.85
Aldrin	1.0E+02	c	0/3	--	0.34	0/3	--	0.34	0/6	--	0.34
alpha-BHC	2.7E+02	c	0/3	--	0.35	0/3	--	0.35	0/6	--	0.35
Alpha-endosulfan (Endosulfan I)	1.2E+05	nc	0/3	--	1.7	0/3	--	1.7	0/6	--	1.7
beta-BHC	9.6E+02	c	0/3	--	0.5	0/3	--	0.5	0/6	--	0.5
Beta-endosulfan (Endosulfan II)	4.1E+06	nc	0/3	--	0.86	0/3	--	0.86	0/6	--	0.86
Chlordane	6.5E+03	c	0/3	--	22	0/3	--	22	0/6	--	22
delta-BHC	1.2E+04	c	0/3	--	0.37	0/3	--	0.37	0/6	--	0.37
Dieldrin	1.1E+02	c	0/3	--	0.48	1/3	0.51 J	0.48	1/6	0.51 J	0.48
Endosulfan sulfate	4.1E+06	nc	0/3	--	0.57	0/3	--	0.57	0/6	--	0.57
Endrin	1.8E+05	nc	0/3	--	0.45	0/3	--	0.45	0/6	--	0.45
Endrin aldehyde	2.0E+05	nc	0/3	--	1.4	0/3	--	1.4	0/6	--	1.4
gamma-BHC (Lindane)	2.1E+03	c	0/3	--	0.45	0/3	--	0.45	0/6	--	0.45
Heptachlor	3.8E+02	c	0/3	--	0.83	0/3	--	0.83	0/6	--	0.83
Heptachlor epoxide	1.9E+02	c	0/3	--	0.39	0/3	--	0.39	0/6	--	0.39
Toxaphene	1.6E+03	c	0/3	--	44	0/3	--	61	0/6	--	61
Aroclors (ug/kg - dry weight)											
Aroclor-1016	2.1E+04	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1221	5.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1232	5.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19

Table 2
Results of Screening of TCRA Soils Collected from the Upland Sand Separation Area West of the Impoundments

Analyte	Screening Level for Industrial Soil [a]		Surface Soil			Subsurface Soil			All Soil		
			FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
Aroclor-1242	7.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1248	7.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1254	7.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1260	7.4E+02	c	0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1262	NV		0/3	--	19	0/3	--	19	0/6	--	19
Aroclor-1268	NV		0/3	--	19	0/3	--	19	0/6	--	19
SVOCs (ug/kg -dw)											
2,4,6-Trichlorophenol	1.6E+05	c	0/3	--	14	0/3	--	14	0/6	--	14
2,4-Dichlorophenol	1.8E+06	nc	0/3	--	10	0/3	--	10	0/6	--	10
2,4-Dimethylphenol	1.2E+07	nc	0/3	--	55	0/3	--	55	0/6	--	55
2,4-Dinitrophenol	1.2E+06	nc	0/3	--	170	0/3	--	170	0/6	--	170
2,4-Dinitrotoluene	5.5E+03	c	0/3	--	15	0/3	--	15	0/6	--	15
2,6-Dinitrotoluene	6.2E+05	nc	0/3	--	20	0/3	--	20	0/6	--	20
2-Chloronaphthalene	8.2E+07	nc	0/3	--	16	0/3	--	16	0/6	--	16
2-Chlorophenol	5.1E+06	nc	0/3	--	20	0/3	--	20	0/6	--	20
2-Nitrophenol	4.1E+05	nc	0/3	--	15	0/3	--	15	0/6	--	15
3,3'-Dichlorobenzidine	3.8E+03	c	0/3	--	37	0/3	--	37	0/6	--	37
4,6-Dinitro-o-cresol (2-Methyl-4,6-dinitrophenol)	4.9E+04	nc	0/3	--	14	0/3	--	14	0/6	--	14
4-Bromophenyl phenyl ether	1.1E+03	c	0/3	--	16	0/3	--	16	0/6	--	16
4-Chlorophenyl phenyl ether	8.0E+02	c	0/3	--	14	0/3	--	14	0/6	--	14
4-Nitrophenol	1.1E+05	nc	0/3	--	180	0/3	--	180	0/6	--	180
Acenaphthene	3.3E+07	nc	2/3	2.8 J	14	1/3	3.4 J	14	3/6	3.4 J	14
Acenaphthylene	3.7E+07	nc	3/3	16.5 J	--	1/3	2.3 J	12	4/6	16.5 J	12
Anthracene	1.7E+08	nc	3/3	19.5 J	--	1/3	9.4	16	4/6	19.5 J	16
Benzo(a) anthracene	2.1E+03	c	3/3	53 J	--	3/3	65	--	6/6	65	--
Benzo(a)pyrene	2.1E+02	c	3/3	67.5	--	3/3	75	--	6/6	75	--
Benzo(b) fluoranthene	2.1E+03	c	3/3	75	--	3/3	92	--	6/6	92	--
Benzo(ghi)perylene	1.9E+07	nc	3/3	91.5	--	2/3	77.5	15	5/6	91.5	15
Benzo(k) fluoranthene	2.1E+04	c	3/3	28.5 J	--	2/3	33	14	5/6	33	14
Bis(2-chloroethyl) ether	1.0E+03	c	0/3	--	19	0/3	--	19	0/6	--	19
Bis(2-chloroisopropyl) ether	2.2E+04	c	0/3	--	26	0/3	--	26	0/6	--	26
Bis(2-chloroethoxy) methane	1.8E+06	nc	0/3	--	15	0/3	--	15	0/6	--	15
Bis(2-ethylhexyl) phthalate	1.2E+05	c	2/3	95 J	7	0/3	--	70	2/6	95 J	70
Butyl benzyl phthalate	9.1E+05	c	0/3	--	32	0/3	--	32	0/6	--	32
Chrysene	2.1E+05	c	3/3	55.5 J	--	2/3	71.5 J	15	5/6	71.5 J	15
Dibenzo(a,h) anthracene	2.1E+02	c	2/3	6.7	15	1/3	11	15	3/6	11	15
Diethyl Phthalate	4.9E+08	nc	1/3	3.4 J	13	0/3	--	13	1/6	3.4 J	13
Dimethyl phthalate	9.3E+05	nc	3/3	75	--	3/3	38 J	--	6/6	75	--
Di-N-Butyl phthalate	6.2E+07	nc	0/3	--	79	0/3	--	79	0/6	--	79
Di-n-octyl phthalate	1.3E+07	nc	1/3	860	1.7	0/3	--	17	1/6	860	17
Fluoranthene	2.2E+07	nc	3/3	84.5	--	3/3	110	--	6/6	110	--
Fluorene	2.2E+07	nc	2/3	2.3 J	11	1/3	2.8 J	11	3/6	2.8 J	11
Hexachlorobenzene	1.1E+03	c	0/3	--	12	0/3	--	12	0/6	--	12

Table 2
Results of Screening of TCRA Soils Collected from the Upland Sand Separation Area West of the Impoundments

Analyte	Screening Level for Industrial Soil [a]		Surface Soil			Subsurface Soil			All Soil		
			FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
Hexachlorobutadiene	2.2E+04	c	0/3	--	25	0/3	--	25	0/6	--	25
Hexachloroethane	3.7E+06	nc	0/3	--	31	0/3	--	31	0/6	--	31
Hexachlorocyclopentadiene	1.2E+05	c	0/3	--	290	0/3	--	290	0/6	--	290
Indeno (1,2,3-cd) pyrene	2.1E+03	c	3/3	68.5	--	2/3	71.5	11	5/6	71.5	11
Isophorone	1.8E+06	c	0/3	--	10	0/3	--	10	0/6	--	10
Naphthalene	1.8E+04	c	0/3	--	23	0/3	--	23	0/6	--	23
Nitrobenzene	2.4E+04	c	0/3	--	22	0/3	--	22	0/6	--	22
N-nitrosodi-n-propylamine	2.5E+02	c	0/3	--	24	0/3	--	24	0/6	--	24
N-nitrosodimethylamine	3.4E+01	c	0/3	--	61	0/3	--	61	0/6	--	61
N-nitrosodiphenylamine	3.5E+05	c	0/3	--	16	0/3	--	16	0/6	--	16
Parachlorometa cresol (4-chloro-3-methylphenol)	6.2E+07	nc	0/3	--	14	0/3	--	14	0/6	--	14
Pentachlorophenol	2.7E+03	c	0/3	--	200	0/3	--	200	0/6	--	200
Phenanthrene	1.9E+07	nc	3/3	49.5 J	--	2/3	43 J	14	5/6	49.5 J	14
Phenol	1.8E+08	nc	0/3	--	20	0/3	--	20	0/6	--	20
Pyrene	1.7E+07	nc	3/3	90	--	3/3	100	--	6/6	100	--
VOCs (ug/kg - dry weight)											
1,1,1-Trichloroethane	3.8E+07	nc	0/3	--	0.42	0/3	--	0.41	0/6	--	0.42
1,1,2,2-Tetrachloroethane	2.8E+03	c	0/3	--	0.43	0/3	--	0.42	0/6	--	0.43
1,1,2-Trichloroethane	5.3E+03	c	0/3	--	0.28	0/3	--	0.27	0/6	--	0.28
1,1-Dichloroethane	1.7E+04	c	0/3	--	0.24	0/3	--	0.24	0/6	--	0.24
1,1-Dichloroethylene	1.1E+06	nc	0/3	--	0.28	0/3	--	0.27	0/6	--	0.28
1,2,4-Trichlorobenzene	9.9E+04	c	0/3	--	0.28	0/3	--	0.27	0/6	--	0.28
1,2-Dichlorobenzene	9.8E+06	nc	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
1,2-Dichloroethane	2.2E+03	c	0/3	--	0.18	0/3	--	0.18	0/6	--	0.18
1,2-Dichloropropane	4.5E+03	c	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
1,2-trans-Dichloroethylene	6.9E+05	nc	0/3	--	0.42	0/3	--	0.41	0/6	--	0.42
1,3-Dichlorobenzene	8.8E+04	nc	0/3	--	0.29	0/3	--	0.28	0/6	--	0.29
1,4-Dichlorobenzene	1.2E+04	c	0/3	--	0.3	0/3	--	0.29	0/6	--	0.3
2-Chloroethyl vinyl ether	3.3E+03	nc	0/3	--	0.58	0/3	--	0.56	0/6	--	0.58
Acrolein	6.5E+02	nc	0/3	--	3.1	0/3	--	3	0/6	--	3.1
Acrylonitrile	1.2E+03	c	0/3	--	1.5	0/3	--	1.4	0/6	--	1.5
Benzene	5.4E+03	c	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
Bromoform	2.2E+05	c	0/3	--	0.36	0/3	--	0.35	0/6	--	0.36
Carbon tetrachloride	3.0E+03	c	0/3	--	0.43	0/3	--	0.42	0/6	--	0.43
Chlorobenzene	1.4E+06	nc	0/3	--	0.3	0/3	--	0.29	0/6	--	0.3
Chlorodibromomethane	3.3E+03	c	0/3	--	0.23	0/3	--	0.22	0/6	--	0.23
Chloroethane	6.1E+07	nc	0/3	--	0.37	0/3	--	0.36	0/6	--	0.37
Chloroform	1.5E+03	c	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
cis-1,3-Dichloropropene	4.3E+04	nc	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
Dichlorobromomethane	1.4E+03	c	0/3	--	0.18	0/3	--	0.18	0/6	--	0.18
Ethylbenzene	2.7E+04	c	0/3	--	0.27	0/3	--	0.26	0/6	--	0.27
Methyl bromide	3.2E+04	nc	0/3	--	0.55	0/3	--	0.54	0/6	--	0.55
Methyl chloride	5.3E+04	c	0/3	--	9.7	0/3	--	8.1	0/6	--	9.7

Table 2
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Analyte		Screening Level for Industrial Soil [a]		Surface Soil			Subsurface Soil			All Soil		
				FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects	FOD	Max Detected	Max Reporting Limit for Nondetects
	Tetrachloroethylene	2.6E+03	c	0/3	--	0.31	0/3	--	0.3	0/6	--	0.31
	Toluene	4.5E+07	nc	0/3	--	0.22	0/3	--	0.21	0/6	--	0.22
	trans-1,3-Dichloropropene	6.1E+04	c	0/3	--	0.41	0/3	--	0.4	0/6	--	0.41
	Trichloroethylene	1.4E+04	c	0/3	--	0.33	0/3	--	0.32	0/6	--	0.33
	Vinyl chloride	1.7E+03	c	0/3	--	0.37	0/3	--	0.36	0/6	--	0.37
Asbestos (%)												
	Asbestos	2.5E-01 [d]	nr	0/3	--	0.25	0/3	--	0.25	0/6	--	0.25

Notes

-- = Not applicable.
c = Screening level is based on carcinogenic endpoint
FOD = frequency of detection
J = Result is less than the method reporting limit but greater than or equal to the method detection limit and the concentration is an estimated value.
nc = Screening level is based on a noncancer endpoint
nr = Screening level is not risk based
RSL = Regional Screening Level
SVOC = semivolatile organic compound
VOC = volatile organic compound

a - Screening levels were selected using the following tiered approach:
Tier 1: USEPA 2010b. USEPA RSLs for Industrial Soil- available at: http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/Generic_Tables/index.htm. Accessed Dec 7, 2010.
Tier 2: TCEQ 2010. TCEQ Tier 1 Commercial/Industrial PCLs for 30 acre source area. Available at: http://www.tceq.state.tx.us/remediation/trrp/trrppcls.html
Values for 1,3-dichlorobenzene, cis-1,3-dichloropropene, trans-1,3-dichloropropene, 2-chloroethyl vinyl ether, 2-nitrophenol, 4-chlorophenyl phenyl ether, 4-nitrophenol, 4-bromophenyl phenyl ether, acenaphthylene, endrin-aldehyde, benzo(ghi)perylene, delta-BHC, carbazole, dimethyl phthalate, di-n-octyl phthalate, alpha-endosulfan, beta-endosulfan, endosulfan-sulfate, phenanthrene, thallium are from TCEQ. The remainder of chemical screening levels are from USEPA (2010b).
Asbestos value based on USEPA 2008. Framework for Investigating Asbestos Contaminated Superfund Sites. OSWER Directive #9200.0-68 September 2008.
b- Value is for chromium(III). Value for chromium(VI) is lower.
c- Value shown is the cancer risk based RSL for industrial/commercial soils from USEPA (2010b). The value of 950 ng/kg dw proposed as a draft preliminary remediation goal (PRG) for industrial/commercial soil (USEPA 2009d) based on a noncancer endpoint should also be considered.
d- Asbestos screening level is not risk based. EPA recommends CARB 435 as a qualitative screening method for determination of the presence or absence of asbestos during the initial stages of a site assessment. This method was used here with the USEPA specified reporting limit of 0.25%.

Table 3
Summary of TEQ_{DF} (ng/kg dw) in Houston Area Soils

Station ID	Sample Soil Type	Date	TEQ_{DF} (ng/kg dw) [a]
SS-5	Soil	9/30/2004	0.57
SS-7	Soil	10/1/2004	1.54
	Soil	10/1/2004	8.6
SS-8	Forest Soil	5/30/2005	8.0
	Grass Soil	5/30/2005	7.7
	Residential Soil	5/30/2005	0.69
	Transitional Soil	5/30/2005	28
SS-9	Soil	10/1/2004	6.5
	Forest Soil	1/27/2005	2.7
	Grass Soil	1/27/2005	2.2
	Residential Soil	1/27/2005	3.4
	Transitional Soil	1/27/2005	3.9
	Urban Soil	1/27/2005	2.52
SS-10	Soil	10/1/2004	0.94
	Forest Soil	12/16/2004	0.59
	Grass Soil	12/16/2004	0.72
	Residential Soil	12/16/2004	0.42
	Transitional Soil	12/16/2004	1.24
	Urban Soil	12/16/2004	1.83
SS-13	Soil	10/1/2004	5.0
	Forest Soil	1/13/2005	0.56
	Grass Soil	1/13/2005	2.45
	Transitional Soil	1/13/2005	0.54
	Urban Soil	1/13/2005	1.58
SS-14	Soil	9/30/2004	0.85
	Forest Soil	3/2/2005	0.41
	Grass Soil	3/2/2005	0.66
	Residential Soil	3/2/2005	1.19
	Transitional Soil	3/2/2005	0.39
	Urban Soil	3/2/2005	0.46
SS-15	Soil	9/30/2004	8.8
	Forest Soil	2/24/2005	1.76
	Grass Soil	2/24/2005	2.67
	Residential Soil	2/24/2005	5.0
	Transitional Soil	2/24/2005	0.95
	Urban Soil	2/24/2005	1.99
SS-16	Soil	9/30/2004	8.9
	Forest Soil	3/15/2005	0.36
	Grass Soil	3/15/2005	0.46
	Residential Soil	3/15/2005	2.16
	Transitional Soil	3/15/2005	1.24
	Urban Soil	3/15/2005	2.28
SS-108	Soil	9/30/2004	1.64

Notes
Use of the hard copy TMDL report (University of Houston and Parsons 2006) as a source of information on environmental samples may generate results that are different from those in the site database and shown here. See Section 3.3 of the RI/FS Work Plan.

DL = detection limit
ND = nondetect
TEF = toxicity equivalency factor
TEQ_{DF} = toxicity equivalent

a - TEQs were calculated with mammalian TEFs (van den Berg et al. 2006) and by setting ND = 1/2 DL.

Table 4
Dioxin and Furans to be Analyzed in Soils

Analyte	CAS Number
Dioxins/furans (ng/kg-dry weight)	
2,3,7,8-Tetrachlorodibenzo-*p* -dioxin	1746-01-6
1,2,3,7,8-Pentachlorodibenzo-*p* -dioxin	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo-*p* -dioxin	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo-*p* -dioxin	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo-*p* -dioxin	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo-*p* -dioxin	35822-46-9
Octachlorodibenzo-*p* -dioxin	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7
Octachlorodibenzofuran	39001-02-0
Total tetrachlorinated dioxins	41903-57-5
Total pentachlorinated dioxins	36088-22-9
Total hexachlorinated dioxins	34465-46-8
Total heptachlorinated dioxins	37871-00-4
Total tetrachlorinated furans	30402-14-3
Total pentachlorinated furans	30402-15-4
Total hexachlorinated furans	55684-94-1
Total heptachlorinated furans	38998-75-3

Table 5

Chemicals of Potential Concern[a]

Chemical of Interest	Primary COPC	Secondary COPC
Dioxins/Furans		
Dioxins and Furans	E, HH	
Metals		
Aluminum	E	
Arsenic	HH	
Barium	E	
Cadmium	E, HH	
Chromium	HH	
Cobalt	E	
Copper	E, HH	
Lead	E	
Magnesium	E	
Manganese	E	
Mercury	E, HH	
Nickel	E, HH	
Thallium		E
Vanadium	E	
Zinc	E, HH	
Polychlorinated Biphenyls		
Polychlorinated Biphenyls		E, HH
Semivolatile Organic Compounds		
Acenaphthene		E
Fluorene		E
Naphthalene		E
Phenanthrene		E
Phenol		E
Pentachlorophenol		E, HH
Hexachlorobenzene		E, HH
Carbazole		E
Bis(2-ethylhexyl)phthalate	E, HH	
2,4-Dichlorophenol		E
2,4,6-Trichlorophenol		E
2,4,5-Trichlorophenol		E
2,3,4,6-Tetrachlorophenol		E, HH

Notes

COPC = chemical of potential concern

E = ecological receptors

HH = human health receptors

a - Volatile organic compounds (VOCs) will not be measured in soil because they were not detected in 98 samples of sediment from the waste impoundments and near vicinity (Tzhone 2010, pers. comm.).

Table 6

Number of Soil Sampling Locations[a]

Sample Group	Sampling Method and Depth	Number of Locations[b]	Sample Locations	Analytes	Study Elements
Site surface soil	Stainless steel shovel, trowel, or spoon 0–6 inches (0–15 cm)	18	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment, fate and transport
Site subsurface soil	Stainless steel shovel, hand auger, or hand corer 6–12 inches (15–30 cm)	18	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site deep subsurface soil	Stainless steel shovel, hand auger, or hand corer 12–24 inches (30–60 cm)	8	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Groundwater well boring soils	Well-boring equipment specified in the Groundwater SAP. All 5-foot intervals, composited across the depth of the sample	3	Within Area 3	Lithology and grain size. Archival for chemistry.	CSM and fate and transport.
Groundwater well boring surface and shallow subsurface soils	Stainless steel shovel, hand auger, or hand corer 0–6 inches (0–15 cm), 6–12 inches (15–30 cm)	3	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site surface soil	Stainless steel shovel, trowel, or spoon 0 - 6 inches (0 - 15 cm)	6	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site subsurface soil	Stainless steel shovel, hand auger, or hand corer 6 –12 inches (15–30 cm)	6	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Background surface soil	Stainless steel shovel, trowel, or spoon 0–6 inches (0–15 cm)	20	Background areas in the vicinity of the site	Primary COPCs, TOC, and grain size	Assessment of background conditions
Background shallow subsurface soil	Stainless steel shovel, hand auger, or hand corer 6–12 inches (15–30 cm)	20	Background areas in the vicinity of the site	Primary COPCs, TOC, and grain size	Assessment of background conditions

Notes

COPC = chemical of potential concern

TOC = total organic carbon

a - Additional samples will be collected south of I-10. The approach, design and sample numbers and purpose are described in Soil SAP Addendum 1.

b - Locations of samples collected for the TCRA are not included in these location counts.

Table 7
Sample Containers, Preservation, and Holding Time Requirements

Matrix	Container [a] Type	Size	Laboratory	Parameter	Preservation	Holding Time	Sample Size [b]
Soil							
	WMG	8 oz.	CAS - Kelso	TOC	4 ± 2°C	28 days	1 g
				Metals	4 ± 2°C	6 months	10 g
				Mercury	4 ± 2°C	28 days	5 g
	WMG	16 oz.	CAS - Kelso	Grain size	4 ± 2°C	6 months	100 g
	WMG	8 oz.	CAS - Houston	Dioxins/furans	4 ± 2°C/Deep frozen (-20°C) [c]/ -10°C [d]	1 year/1 year [e]	50 g
	WMG	8 oz.	CAS - Houston	PCBs	4 ± 2°C/Deep frozen (-20°C) [c]/ -10°C [d]	1 year/1 year [e]	50 g
	WMG	8 oz.	CAS - Kelso	SVOC/Archival	4 ± 2°C/ Deep frozen (-20°C) [c]	1 year [f]	50 g
Equipment Filter Wipe Blanks							
	WMG	4 oz.	CAS - Kelso	Metals	4 ± 2°C	6 months	1 wipe
	WMG	4 oz.	CAS - Kelso	Mercury	4 ± 2°C	28 days	1 wipe
	WMG	4 oz.	CAS - Houston	Dioxins/furans	4 ± 2°C	1 year/1 year [e]	1 wipe
	WMG	4 oz.	CAS - Houston	PCBs	4 ± 2°C	1 year/1 year [e]	1 wipe
	WMG	4 oz.	CAS - Kelso	SVOCs	4 ± 2°C	14 days/40 days [e]	1 wipe

Notes

PCB = polychlorinated biphenyl
SVOC = semivolatile organic compound
TOC = total organic carbon
WMG = wide mouth glass
a - The size and number of containers may be modified by the analytical laboratory.
b - Sample sizes are estimated.
c - Samples will be shipped to the laboratory on ice at 4 ± 2°C. Once received at the laboratory, samples will be stored at -20°C.
d - Extracts will be stored at -10 °C.
e - Holding time for samples prior to extraction/holding time for extracts.
f - Holding time for frozen samples is 1 year.

Table 8
Proposed Laboratory Methods for Soil Samples

Parameter	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Conventional and Geotechnical					
Percent moisture	CAS-Kelso	USEPA 160.3	Oven drying	USEPA 160.3	Balance/gravimetric
Total organic carbon [a]	CAS-Kelso	USEPA 9060A	Acid pretreatment	EPA 9060A (modified for sediment)	Combustion
		Walkley - Black	dichromate oxidation	Walkley - Black	Titration
Grain size	CAS-Kelso	NA	NA	ASTM D-422 and D-1140 with USEPA (1986) modifications	Sieves and pipette method
Metals					
Arsenic, cadmium, chromium	CAS-Kelso	USEPA 3050	Strong acid digestion	USEPA 6020	ICP/MS
Aluminum, barium, cobalt, copper, lead, magnesium, manganese, nickel, thallium, vanadium, zinc	CAS-Kelso	USEPA 3050	Strong acid digestion	USEPA 6010B	ICP
Mercury	CAS-Kelso	USEPA 7471A	Acid digestion/oxidation	USEPA 7471A	CVAA
Organics					
Dioxins /furans	CAS-Houston	USEPA 1613B	Soxhlet extraction	USEPA 1613B	HRGC/HRMS
			Silica gel column cleanup		
			Additional cleanup as needed		
PCB Congeners	CAS-Houston	USEPA 1668A	Soxhlet extraction	USEPA 1668A	HRGC/HRMS
			Silica gel column cleanup		
			Additional cleanup as needed		

Table 8
Proposed Laboratory Methods for Soil Samples

Parameter	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Conventional and Geotechnical					
SVOCs	CAS-Kelso	USEPA 3540C/3541/ 3545A	Soxhlet/automated Soxhlet/pressurized fluid extraction	USEPA 8270C	GC/MS
		USEPA 3640A	Gel permeation chromatography		

Notes

ASTM = American Society for Testing and Materials
CVAA = cold vapor atomic absorbtion spectrometry
GC/MS = gas chromatography/mass spectrometry
HRGC = high-resolution gas chromatography
HRMS = high-resolution mass spectrometry
ICP = inductively coupled plasma-atomic emission spectrometry
ICP/MS = inductively coupled plasma/mass spectrometry
NA = not applicable
SVOC = semivolatile organic compound
USEPA = U.S. Environmental Protection Agency

a - TOC will be determined by the Walkley-Black method for groundwater well boring soils. TOC for all other soils will be determined by USEPA method 9060A.

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
Conventionals					
Percent moisture (percent)	--	NA	NA	NA	NA
Total organic carbon (percent)	--	NA	NA	0.02	0.05
Metals (mg/kg-dry weight)					
Aluminum	7429-90-5	77,000	NA[c]	6	10
Arsenic	7440-38-2	0.39	18	0.06	0.5
Barium	7440-39-3	15,000	330	0.3	2
Cadmium	7440-43-9	70	0.36	0.004	0.02
Chromium	7440-47-3	120,000	26	0.03	0.2
Cobalt	7440-48-4	23	13	0.3	2
Copper	7440-50-8	3,100	28	0.6	2
Lead	7439-92-1	400	11	3	20
Magnesium	7439-95-4	--	NA	0.04	4
Manganese	7439-96-5	1,800	220	0.04	2
Nickel	7440-02-0	1,500	38.00	0.5	4
Thallium	7440-28-0	6.3	NA	3	20
Vanadium	7440-62-2	390	7.8	0.4	2
Zinc	7440-66-6	23,000	46	0.3	2
Mercury	7439-97-6	23	NA	0.002	0.02
Organics					
Dioxins/furans (ng/kg-dry weight)					
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	35822-46-9	--	NA	0.0539	5
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	--	NA	0.0482	5
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	--	NA	0.0561	5
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	39227-28-6	--	NA	0.0616	5
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	--	NA	0.0688	5
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	57653-85-7	--	NA	0.0500	5
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	--	NA	0.0489	5
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	19408-74-3	--	NA	0.0525	5

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	--	NA	0.0521	5
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	--	NA	0.0501	5
1,2,3,7,8-Pentachlorodibenzo-*p* -dioxin	40321-76-4	--	NA	0.0656	5
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	--	NA	0.0490	5
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	--	NA	0.0444	5
2,3,7,8-Tetrachlorodibenzo-*p* -dioxin	1746-01-6	4.5	NA	0.0664	1
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	--	NA	0.0726	1
Octachlorodibenzo-*p* -dioxin	3268-87-9	--	NA	0.0990	10
Octachlorodibenzofuran	39001-02-0	--	NA	0.0782	10
Total tetrachlorinated dioxins	41903-57-5	--	NA	NA	1
Total pentachlorinated dioxins	36088-22-9	--	NA	NA	5
Total hexachlorinated dioxins	34465-46-8	--	NA	NA	5
Total heptachlorinated dioxins	37871-00-4	--	NA	NA	5
Total tetrachlorinated furans	30402-14-3	--	NA	NA	1
Total pentachlorinated furans	30402-15-4	--	NA	NA	5
Total hexachlorinated furans	55684-94-1	--	NA	NA	5
Total heptachlorinated furans	38998-75-3	--	NA	NA	5
2,3,7,8-TCDD TEQ	NA	4.5	--	NA	NA
PCB Congeners, dioxin-like (ng/kg-dry weight)					
3,3'-4,4'-TetraCB (77)	32598-13-3	34,000	NA	85	250
3,4,4',5-TetraCB (81)	70362-50-4	11,000	NA	90	250
2,3,3',4,4'-PentaCB (105)	32598-14-4	110,000	NA	55	100
2,3,4,4',5-PentaCB (114)	74472-37-0	110,000	NA	60	250
2,3',4,4',5-PentaCB (118)	31508-00-6	110,000	NA	95	250
2',3,4,4',5-PentaCB (123)	65510-44-3	110,000	NA	75	250
3,3',4,4',5-PentaCB (126)	57465-28-8	34	NA	70	250
2,3,3',4,4',5-HexaCB (156)	38380-08-4	110,000	NA	65	250
2,3,3',4,4',5'-HexaCB (157)	69782-90-7	110,000	NA	65	250
2,3',4,4',5,5'-HexaCB (167)	52663-72-6	110,000	NA	55	250

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
3,3',4,4',5,5'-HexaCB (169)	32774-16-6	110	NA	80	250
2,3,3',4,4',5,5'-HeptaCB (189)	39635-31-9	110,000	NA	90	250
PCB Congeners (ng/kg-dry weight)					
2-MonoCB (1)	2051-60-7	--	NA	40	100
3-MonoCB (2)	2051-61-8	--	NA	2	5
4-MonoCB (3)	2051-62-9	--	NA	45	100
2,2'-DiCB (4)	13029-08-8	--	NA	85	250
2,3-DiCB (5)	16605-91-7	--	NA	5	25
2,3'-DiCB (6)	25569-80-6	--	NA	5	25
2,4-DiCB (7)	33284-50-3	--	NA	10	25
2,4'-DiCB (8)	34883-43-7	--	NA	60	250
2,5-DiCB (9)	34883-39-1	--	NA	10	25
2,6-DiCB (10)	33146-45-1	--	NA	10	25
3,3'-DiCB (11)	2050-67-1	--	NA	50	500
3,4-DiCB (12)	2974-92-7	--	NA	15	50
3,4'-DiCB (13)	2974-90-5	--	NA	15	50
3,5-DiCB (14)	34883-41-5	--	NA	15	50
4,4'-DiCB (15)	2050-68-2	--	NA	90	250
2,2',3-TriCB (16)	38444-78-9	--	NA	20	50
2,2',4-TriCB (17)	37680-66-3	--	NA	45	100
2,2',5-TriCB (18)	37680-65-2	--	NA	100	250
2,2',6-TriCB (19)	38444-73-4	--	NA	20	50
2,3,3'-TriCB (20)	38444-84-7	--	NA	95	250
2,3,4-TriCB (21)	55702-46-0	--	NA	25	100
2,3,4'-TriCB (22)	38444-85-8	--	NA	45	100
2,3,5-TriCB (23)	55720-44-0	--	NA	25	100
2,3,6-TriCB (24)	55702-45-9	--	NA	25	100
2,3',4-TriCB (25)	55712-37-3	--	NA	25	100
2,3',5-TriCB (26)	38444-81-4	--	NA	40	100

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,3',6-TriCB (27)	38444-76-7	--	NA	30	100
2,4,4'-TriCB (28)	7012-37-5	--	NA	95	250
2,4,5-TriCB (29)	15862-07-4	--	NA	40	100
2,4,6-TriCB (30)	35693-92-6	--	NA	100	250
2,4',5-TriCB (31)	16606-02-3	--	NA	75	250
2,4',6-TriCB (32)	38444-77-8	--	NA	40	100
2',3,4-TriCB (33)	38444-86-9	--	NA	25	100
2',3,5-TriCB (34)	37680-68-5	--	NA	35	100
3,3',4-TriCB (35)	37680-69-6	--	NA	40	100
3,3',5-TriCB (36)	38444-87-0	--	NA	40	100
3,4,4'-TriCB (37)	38444-90-5	--	NA	65	250
3,4,5-TriCB (38)	53555-66-1	--	NA	40	100
3,4',5-TriCB (39)	38444-88-1	--	NA	45	100
2,2',3,3'-TetraCB (40)	38444-93-8	--	NA	60	250
2,2',3,4-TetraCB (41)	52663-59-9	--	NA	60	250
2,2',3,4'-TetraCB (42)	36559-22-5	--	NA	30	100
2,2',3,5-TetraCB (43)	70362-46-8	--	NA	45	250
2,2',3,5'-TetraCB (44)	41464-39-5	--	NA	95	250
2,2',3,6-TetraCB (45)	70362-45-7	--	NA	25	100
2,2',3,6'-TetraCB (46)	41464-47-5	--	NA	50	100
2,2',4,4'-TetraCB (47)	2437-79-8	--	NA	95	250
2,2',4,5-TetraCB (48)	70362-47-9	--	NA	40	100
2,2',4,5'-TetraCB (49)	41464-40-8	--	NA	55	250
2,2',4,6-TetraCB (50)	62796-65-0	--	NA	30	100
2,2',4,6'-TetraCB (51)	68194-04-7	--	NA	25	100
2,2',5,5'-TetraCB (52)	35693-99-3	--	NA	95	250
2,2',5,6'-TetraCB (53)	41464-41-9	--	NA	30	100
2,2',6,6'-TetraCB (54)	15968-05-5	--	NA	60	250
2,3,3',4-TetraCB (55)	74338-24-2	--	NA	60	250

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,3,3',4'-TetraCB (56)	41464-43-1	--	NA	50	100
2,3,3',5-TetraCB (57)	74472-33-6	--	NA	60	250
2,3,3',5'-TetraCB (58)	41464-49-7	--	NA	65	250
2,3,3',6-TetraCB (59)	74472-33-6	--	NA	30	100
2,3,4,4'-TetraCB (60)	33025-41-1	--	NA	65	250
2,3,4,5-TetraCB (61)	33284-53-6	--	NA	85	250
2,3,4,6-TetraCB (62)	54230-22-7	--	NA	30	100
2,3,4',5-TetraCB (63)	74472-34-7	--	NA	70	250
2,3,4',6-TetraCB (64)	52663-58-8	--	NA	35	100
2,3,5,6-TetraCB (65)	33284-54-7	--	NA	95	250
2,3',4,4'-TetraCB (66)	32598-10-0	--	NA	80	250
2,3',4,5-TetraCB (67)	73575-53-8	--	NA	75	250
2,3',4,5'-TetraCB (68)	73575-52-7	--	NA	75	250
2,3',4,6-TetraCB (69)	60233-24-1	--	NA	55	250
2,3',4',5-TetraCB (70)	32598-11-1	--	NA	85	250
2,3',4',6-TetraCB (71)	41464-46-4	--	NA	60	250
2,4',5,5'-TetraCB (72)	41464-42-0	--	NA	80	250
2,3',5',6-TetraCB (73)	74338-23-1	--	NA	45	250
2,4,4',5-TetraCB (74)	32690-93-0	--	NA	85	250
2,4,4',6-TetraCB (75)	32598-12-2	--	NA	30	100
2',3,4,5-TetraCB (76)	70362-48-0	--	NA	85	250
3,3',4,5-TetraCB (78)	70362-49-1	--	NA	85	250
3,3',4,5'-TetraCB (79)	41464-48-6	--	NA	85	250
3,3',5,5'-TetraCB (80)	33284-52-5	--	NA	90	250
2,2',3,3',4-PentaCB (82)	52663-62-4	--	NA	65	250
2,2',3,3',5-PentaCB (83)	60145-20-2	--	NA	110	250
2,2',3,3',6-PentaCB (84)	52663-60-2	--	NA	60	250
2,2',3,4,4'-PentaCB (85)	65510-45-4	--	NA	50	100
2,2',3,4,5-PentaCB (86)	55312-69-1	--	NA	75	250

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,2',3,4,5'-PentaCB (87)	38380-02-8	--	NA	75	250
2,2',3,4,6-PentaCB (88)	55215-17-3	--	NA	60	250
2,2',3,4,6'-PentaCB (89)	73575-57-2	--	NA	95	250
2,2',3,4',5-PentaCB (90)	68194-07-0	--	NA	120	500
2,2',3,4',6-PentaCB (91)	68194-05-8	--	NA	60	250
2,2',3,5,5'-PentaCB (92)	52663-61-3	--	NA	60	250
2,2',3,5,6-PentaCB (93)	73575-56-1	--	NA	110	250
2,2',3,5,6'-PentaCB (94)	73575-55-0	--	NA	60	250
2,2',3,5',6-PentaCB (95)	38379-99-6	--	NA	110	250
2,2',3,6,6'-PentaCB (96)	73575-54-9	--	NA	105	250
2,2',3',4,5-PentaCB (97)	41464-51-1	--	NA	75	250
2,2',3',4,6-PentaCB (98)	60233-25-2	--	NA	110	250
2,2',4,4',5-PentaCB (99)	38380-01-7	--	NA	110	250
2,2',4,4',6-PentaCB (100)	39485-83-1	--	NA	110	250
2,2',4,5,5'-PentaCB (101)	37680-73-2	--	NA	120	500
2,2',4,5,6'-PentaCB (102)	68194-06-9	--	NA	110	250
2,2',4,5',6-PentaCB (103)	60145-21-3	--	NA	115	250
2,2',4,6,6'-PentaCB (104)	56558-16-8	--	NA	115	250
2,3,3',4,5-PentaCB (106)	70424-69-0	--	NA	70	250
2,3,3',4',5-PentaCB (107)	70424-68-9	--	NA	50	100
2,3,3',4,5'-PentaCB (108)	70362-41-3	--	NA	135	500
2,3,3',4,6-PentaCB (109)	74472-35-8	--	NA	75	250
2,3,3',4',6-PentaCB (110)	38380-03-9	--	NA	120	500
2,3,3',5,5'-PentaCB (111)	39635-32-0	--	NA	120	500
2,3,3',5,6-PentaCB (112)	74472-36-9	--	NA	125	500
2,3,3',5',6-PentaCB (113)	68194-10-5	--	NA	120	500
2,3,4,4',6-PentaCB (115)	74472-38-1	--	NA	120	500
2,3,4,5,6-PentaCB (116)	18259-05-7	--	NA	50	100
2,3,4',5,6-PentaCB (117)	68194-11-6	--	NA	50	100

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,3',4,4',6-PentaCB (119)	56558-17-9	--	NA	75	250
2,3',4,5,5'-PentaCB (120)	68194-12-7	--	NA	75	250
2,3',4,5',6-PentaCB (121)	56558-18-0	--	NA	105	250
2',3,3',4,5-PentaCB (122)	76842-07-4	--	NA	60	250
2',3,4,5,5'-PentaCB (124)	70424-70-3	--	NA	135	500
2',3,4,5,6'-PentaCB (125)	74472-39-2	--	NA	75	250
3,3',4,5,5'-PentaCB (127)	39635-33-1	--	NA	140	500
2,2',3,3',4,4'-HexaCB (128)	38380-07-3	--	NA	60	250
2,2',3,3',4,5-HexaCB (129)	55215-18-4	--	NA	105	250
2,2',3,3',4,5'-HexaCB (130)	52663-66-8	--	NA	70	250
2,2',3,3',4,6-HexaCB (131)	61798-70-7	--	NA	60	250
2,2',3,3',4,6'-HexaCB (132)	38380-05-1	--	NA	60	250
2,2',3,3',5,5'-HexaCB (133)	35694-04-3	--	NA	85	250
2,2',3,3',5,6-HexaCB (134)	52704-70-8	--	NA	65	250
2,2',3,3',5,6'-HexaCB (135)	52744-13-5	--	NA	55	250
2,2',3,3',6,6'-HexaCB (136)	38411-22-2	--	NA	45	100
2,2',3,4,4',5-HexaCB (137)	35694-06-5	--	NA	150	500
2,2',3,4,4',5'-HexaCB (138)	35065-28-2	--	NA	105	250
2,2',3,4,4',6-HexaCB (139)	56030-56-9	--	NA	100	250
2,2',3,4,4',6'-HexaCB (140)	59291-64-4	--	NA	100	250
2,2',3,4,5,5'-HexaCB (141)	52712-04-6	--	NA	45	100
2,2',3,4,5,6-HexaCB (142)	41411-61-4	--	NA	155	500
2,2',3,4,5,6'-HexaCB (143)	68194-15-0	--	NA	65	250
2,2',3,4,5',6-HexaCB (144)	68194-14-9	--	NA	85	250
2,2',3,4,6,6'-HexaCB (145)	74472-40-5	--	NA	160	500
2,2',3,4',5,5'-HexaCB (146)	51908-16-8	--	NA	90	250
2,2',3,4',5,6-HexaCB (147)	68194-13-8	--	NA	90	250
2,2',3,4',5,6'-HexaCB (148)	74472-41-6	--	NA	160	500
2,2',3,4',5',6-HexaCB (149)	38380-04-0	--	NA	90	250

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,2',3,4',6,6'-HexaCB (150)	68194-08-1	--	NA	165	500
2,2',3,5,5',6-HexaCB (151)	52663-63-5	--	NA	55	250
2,2',3,5,6,6'-HexaCB (152)	68194-09-2	--	NA	120	500
2,2',4,4',5,5'-HexaCB (153)	35065-27-1	--	NA	65	250
2,2',4,4',5,6'-HexaCB (154)	60145-22-4	--	NA	55	250
2,2',4,4',6,6'-HexaCB (155)	33979-03-2	--	NA	170	500
2,3,3',4,4',6-HexaCB (158)	74472-42-7	--	NA	50	100
2,3,3',4,5,5'-HexaCB (159)	39635-35-3	--	NA	175	500
2,3,3',4,5,6-HexaCB (160)	41411-62-5	--	NA	105	250
2,3,3',4,5',6-HexaCB (161)	74472-43-8	--	NA	175	500
2,3,3',4',5,5'-HexaCB (162)	39635-34-2	--	NA	175	500
2,3,3',4',5,6-HexaCB (163)	74472-44-9	--	NA	105	250
2,3,3',4',5',6-HexaCB (164)	74472-45-0	--	NA	70	500
2,3,3',5,5',6-HexaCB (165)	74472-46-1	--	NA	180	500
2,3,4,4',5,6-HexaCB (166)	41411-63-6	--	NA	60	250
2,3',4,4',5',6-HexaCB (168)	59291-65-5	--	NA	65	250
2,2',3,3',4,4',5-HeptaCB (170)	35065-30-6	--	NA	80	250
2,2',3,3',4,4',6-HeptaCB (171)	52663-71-5	--	NA	185	500
2,2',3,3',4,5,5'-HeptaCB (172)	52663-74-8	--	NA	190	500
2,2',3,3',4,5,6-HeptaCB (173)	68194-16-1	--	NA	185	500
2,2',3,3',4,5,6'-HeptaCB (174)	38411-25-5	--	NA	95	250
2,2',3,3',4,5',6-HeptaCB (175)	40186-70-7	--	NA	190	500
2,2',3,3',4,6,6'-HeptaCB (176)	52663-65-7	--	NA	195	500
2,2',3,3',4',5,6-HeptaCB (177)	52663-70-4	--	NA	70	250
2,2',3,3',5,5',6-HeptaCB (178)	52663-67-9	--	NA	110	250
2,2',3,3',5,6,6'-HeptaCB (179)	52663-64-6	--	NA	115	250
2,2',3,4,4',5,5'-HeptaCB (180)	35065-29-3	--	NA	70	250
2,2',3,4,4',5,6-HeptaCB (181)	74472-47-2	--	NA	200	500
2,2',3,4,4',5,6'-HeptaCB (182)	60145-23-5	--	NA	200	500

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
2,2',3,4,4',5',6-HeptaCB (183)	52663-69-1	--	NA	200	500
2,2',3,4,4',6,6'-HeptaCB (184)	74472-48-3	--	NA	200	500
2,2',3,4,5,5',6-HeptaCB (185)	52712-05-7	--	NA	200	500
2,2',3,4,5,6,6'-HeptaCB (186)	74472-49-4	--	NA	205	500
2,2',3,4',5,5',6-HeptaCB (187)	52663-68-0	--	NA	95	250
2,2',3,4',5,6,6'-HeptaCB (188)	74487-85-7	--	NA	115	250
2,3,3',4,4',5,6-HeptaCB (190)	41411-64-7	--	NA	115	250
2,3,3',4,4',5',6-HeptaCB (191)	74472-50-7	--	NA	210	500
2,3,3',4,5,5',6-HeptaCB (192)	74472-51-8	--	NA	210	500
2,3,3',4',5,5',6-HeptaCB (193)	69782-91-8	--	NA	70	250
2,2',3,3',4,4',5,5'-OctaCB (194)	65694-08-7	--	NA	85	250
2,2',3,3',4,4',5,6-OctaCB (195)	52663-78-2	--	NA	215	500
2,2',3,3',4,4',5,6'-OctaCB (196)	42740-50-1	--	NA	215	500
2,2',3,3',4,4',6,6'-OctaCB (197)	33091-17-7	--	NA	125	500
2,2',3,3',4,5,5',6-OctaCB (198)	68194-17-2	--	NA	100	250
2,2',3,3',4,5,5',6'-OctaCB (199)	52663-75-9	--	NA	100	250
2,2',3,3',4,5,6,6'-OctaCB (200)	52663-73-7	--	NA	125	500
2,2',3,3',4,5',6,6'-OctaCB (201)	40186-71-8	--	NA	220	500
2,2',3,3',5,5',6,6'-OctaCB (202)	2136-99-4	--	NA	220	500
2,2',3,4,4',5,5',6-OctaCB (203)	52663-76-0	--	NA	220	500
2,2',3,4,4',5,6,6'-OctaCB (204)	74472-52-9	--	NA	225	500
2,3,3',4,4',5,5',6-OctaCB (205)	74472-53-0	--	NA	225	500
2,2',3,3',4,4',5,5',6-NonaCB (206)	40186-72-9	--	NA	225	500
2,2',3,3',4,4',5,6,6'-NonaCB (207)	52663-79-3	--	NA	225	500
2,2',3,3',4,5,5',6,6'-NonaCB (208)	52663-77-1	--	NA	230	500
2,2',3,3',4,4',5,5',6,6'-DecaCB (209)	2051-24-3	--	NA	75	250
Semivolatile Organic Compounds (µg/kg-dry weight)					
Acenaphthene	83-32-9	3,400,000	NA	1.4	10
Fluorene	86-73-7	2,300,000	NA	1.1	10

Table 9
Analytes, Analytical Concentration Goals, Method Reporting Limits, and Method Detection Limits for Soil Samples

Analyte	CAS Number	HHRA ACG [a]	BERA ACG [b]	Method Detection Limit	Method Reporting Limit
Naphthalene	91-20-3	3,600	NA	2.3	10
Phenanthrene	85-01-8	1,700,000	NA	1.4	10
2,4,6-Trichlorophenol	88-06-2	44,000	NA	1.4	10
2,4-Dichlorophenol	120-83-2	180,000	NA	1.0	10
Pentachlorophenol	87-86-5	890	2,100	20	100
Phenol	108-95-2	18,000,000	NA	2.0	30
Hexachlorobenzene	118-74-1	300	NA	1.2	10
2,3,4,6-Tetrachlorophenol	58-90-2	1,800,000	NA	--	--
Carbazole	86-74-8	230,000	NA	1.3	10
2,4,5-Trichlorophenol	95-95-4	6,100,000	NA	1.5	10
Bis(2-ethylhexyl)phthalate	117-81-7	35,000	NA	7.0	100

Notes

ACG = analytical concentration goal
BERA = baseline ecological risk assessment
HHRA = human health risk assessment
NA = not applicable
NOEC = no observed effect concentration
PCL = protective concentration level
PRG = preliminary remediation goal
TBD = to be determined
TCEQ = Texas Commission on Environmental Quality
TEQ = toxicity equivalent
TRRP = Texas Risk Reduction Program
USEPA = U.S. Environmental Protection Agency

-- = information not available

a - HHRA ACGs were selected using a tierd approach:
Tier 1: USEPA Regional Screening Levels for Residential Soil (USEPA 2010b).
Tier 2: TRRP PCLs for residential soils selected for these analytes (TCEQ 2010).
Values shown are Tier 1 with the exception of thallium, phenanthrene, and carbazole. These were obtained from TCEQ (2010).

b - BERA ACGs are the Ecological Soil Screening Level (EcoSSL) values (USEPA 2005a). The HHRA ACG is used when no NOEC value is available.

c - USEPA considers aluminum to be a COPC for ecological receptors only in soils with a soil pH lower than 5.5 (USEPA 2002c).

FIGURES

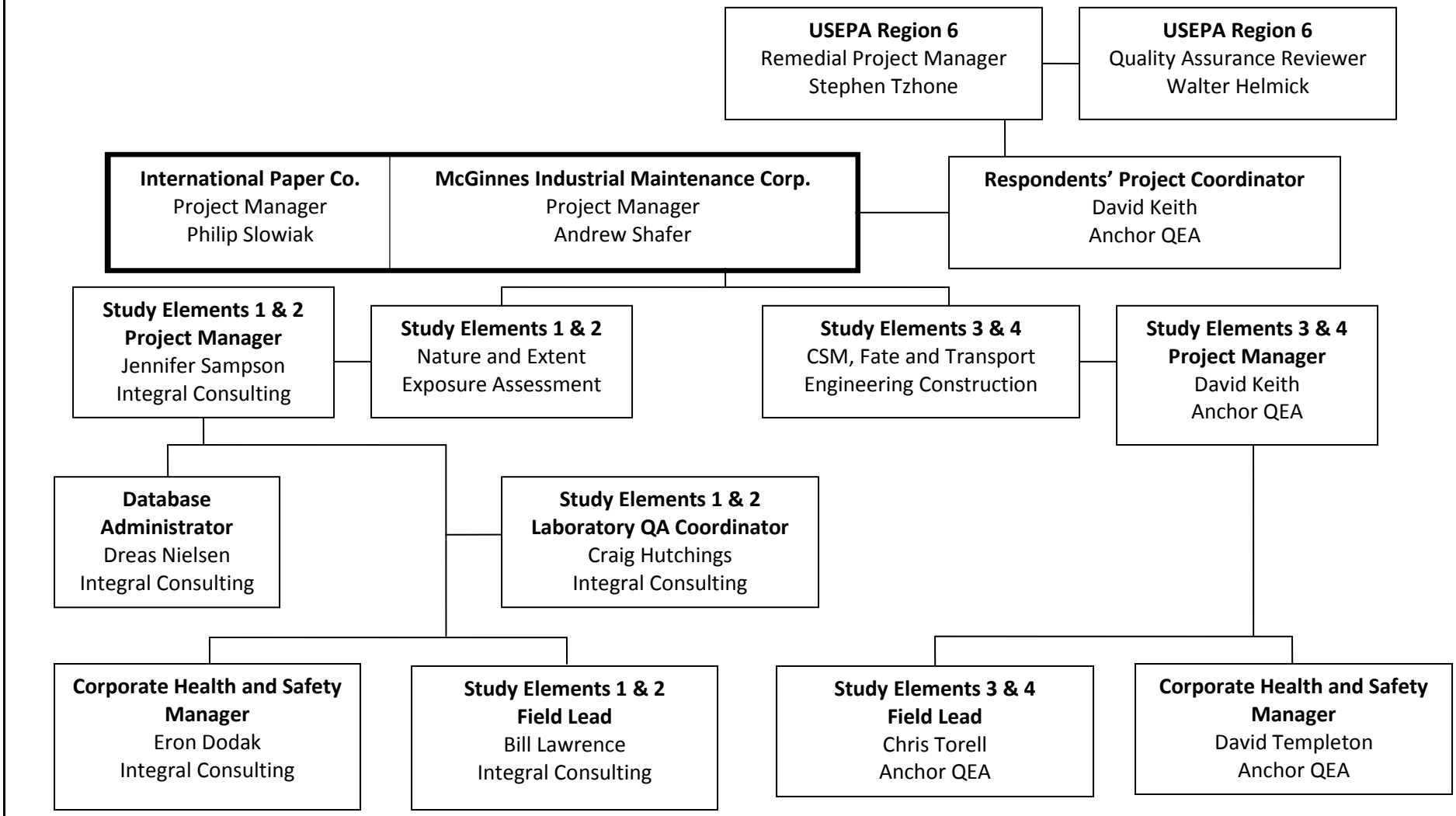

Figure 1
Project Organization Chart
SJRWP Soil SAP
SJRWP Superfund/MIMC and IPC

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ANCHOR QEA

integral consulting inc

0 800

Scale in Feet

- Mean High Water (+1 ft NAVD88)
- USEPA's Preliminary Site Perimeter
- Original (1966) Perimeter of the Northern Impoundments
- Area of Soil Investigation South of I-10

[a] Designation of the sand separation area is intended to be a general reference to areas in which such activities are believed to have taken place based on visual observations of aerial photography from 1998 through 2002.

FEATURE SOURCES:
Aerial Imagery: 0.5-meter. Photo Date: 01/14/2009
Texas Strategic Mapping Program (StratMap), TNRIS

Figure 2
Overview of Soil Study Area
SJRWP Soil SAP
SJRWP Superfund/MIMC and IPC

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ANCHOR QEA

integral consulting inc.

0 400

Scale in Feet

FEATURE SOURCES:
Aerial Imagery: 0.5-meter 2008/2009 DOQQs-
Texas Strategic Mapping Program (StratMap)

- Groundwater well/soil sampling station
- TCRA Soil Sampling Location Upland Sand Separation Area
- TCRA Soil Sampling Location, TxDOT ROW
- RI/FS Sediment Station
- USEPA's Preliminary Site Perimeter
- Soil Sampling Location (0-6 and 6-12 in)
- Soil Sampling Location (12-24 in)
- Proposed Contractor Access and Laydown Area
- Mean High Water (+1 ft NAVD88)

Figure 3
Soil Sample Locations
for the Area North of I-10
SJRWP Soil SAP
SJRWP Superfund/MIMC and IPC

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integral consulting inc.

Soil Sample Locations

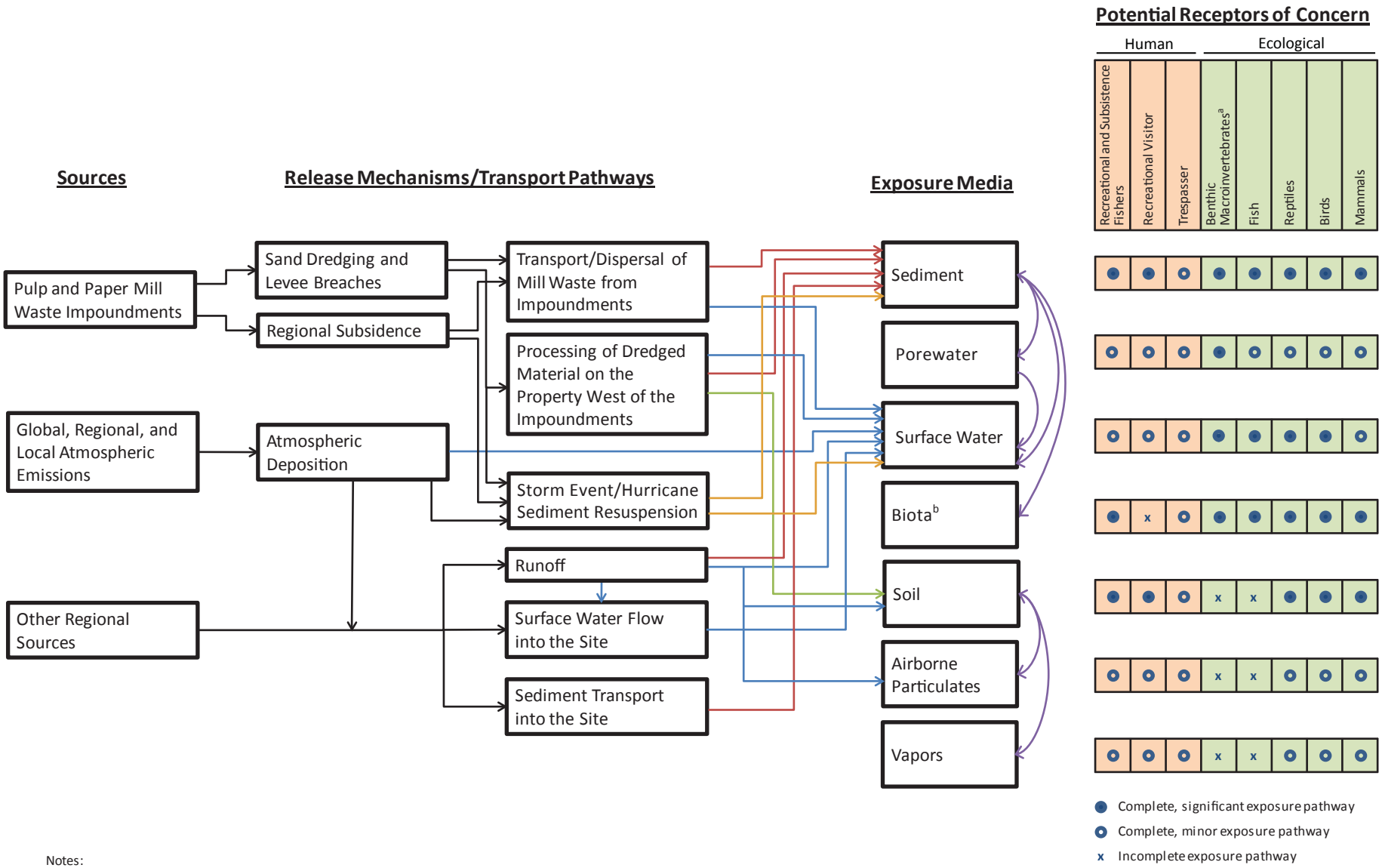
Preliminary Site Perimeter

0 400

Feet

FEATURE SOURCES:
Aerial Imagery: 0.5-meter 2008/2009 DOQQs-
Texas Strategic Mapping Program (StratMap)

Figure 4
TEQ Concentrations in Soil (pg/g)
SJRWP Soil SAP
SJRWP Superfund/MIMC and IPC



Notes:

Other regional sources may include industrial effluents, publicly owned treatment works, and stormwater.

Curved lines indicate potential transport pathways for chemicals of potential concern among exposure media.

[a]Benthic invertebrates include crabs and other crustaceans and shellfish consumed by all receptors, as well as polychaetes and other infauna consumed by fish, other marine life, birds and mammals.

[b]Biota consumed by human receptors are expected to be fish and shellfish.

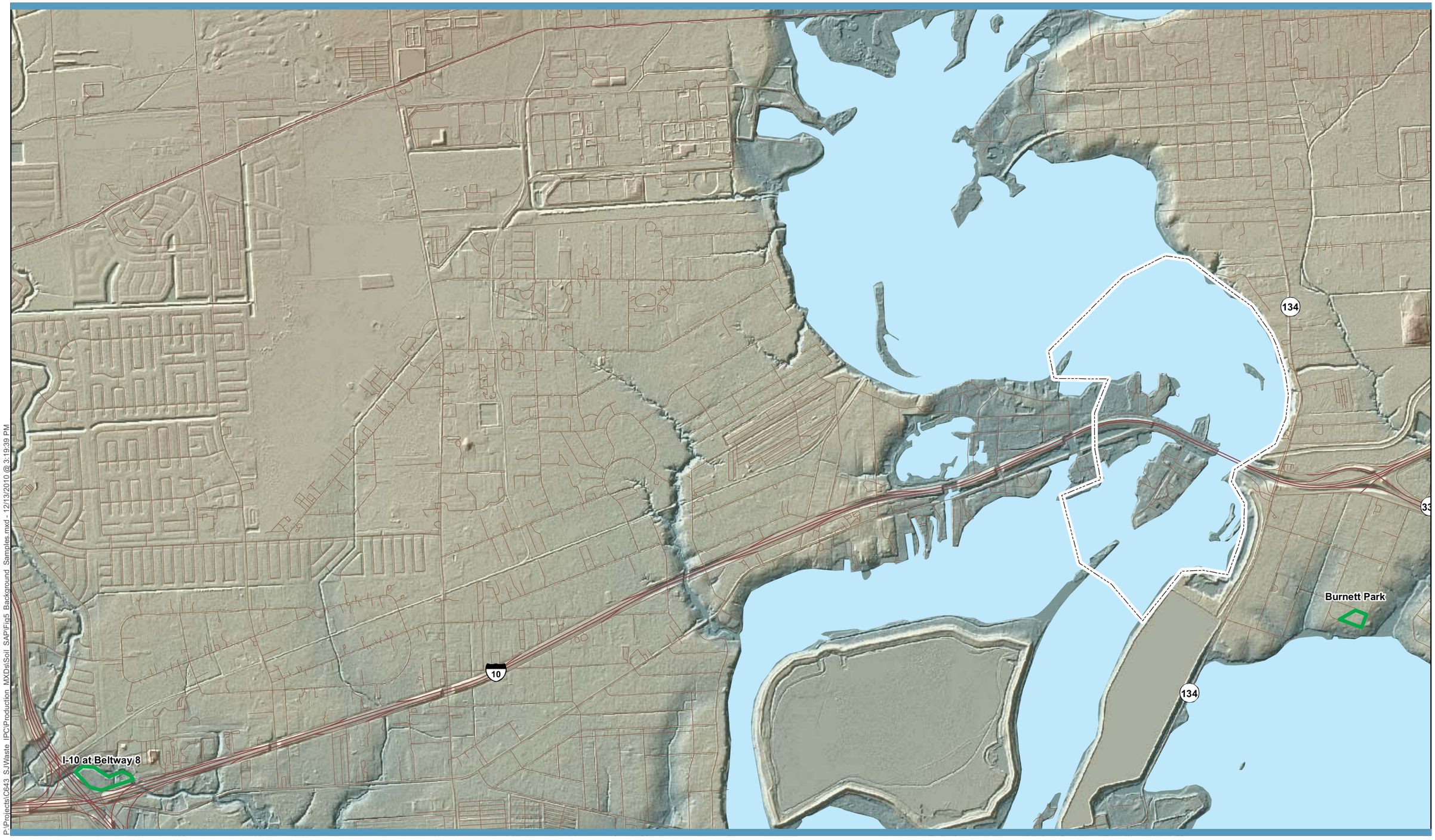
ANCHOR QEA integral consulting inc.

Figure 5

CSM Pathway Diagram for the Area North of I-10

SJRWP Soil SAP

SJRWP Superfund/MIMC and IPC



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Scale in Feet

USEPA's Preliminary Site Perimeter

County Park

Figure 6
Background Soil Sampling Locations
SJRWP Soil SAP
SJRWP Superfund/MIMC and IPC

APPENDIX A
SOIL FIELD SAMPLING PLAN
SAN JACINTO RIVER WASTE PITS SUPERFUND SITE

SOIL FIELD SAMPLING PLAN
SAN JACINTO RIVER WASTE PITS SUPERFUND SITE

Prepared for

McGinnes Industrial Maintenance Corporation
International Paper Company
U.S. Environmental Protection Agency, Region 6

Prepared by

Integral Consulting Inc.
411 First Avenue South, Suite 550
Seattle, Washington 98104

December 2010

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LIST OF ACRONYMS AND ABBREVIATIONS

Abbreviation	Definition
Anchor QEA	Anchor QEA, LLC
ASTM	American Society for Testing and Materials
COC	chain-of-custody
COPC	chemical of potential concern
DGPS	differential global positioning system
FSP	Field Sampling Plan
GPS	global positioning system
HASP	Health and Safety Plan
I-10	Interstate Highway 10
Integral	Integral Consulting Inc.
IPC	International Paper Company
MIMC	McGinnes Industrial Maintenance Corporation
PCB	polychlorinated biphenyl
QA	quality assurance
QA/QC	quality assurance and quality control
QC	quality control
RI/FS	Remedial Investigation and Feasibility Study
SAP	Sampling and Analysis Plan
Site	San Jacinto River Waste Pits Superfund Site
SJRWP	San Jacinto River Waste Pits
SOP	standard operating procedure
SVOC	semivolatile organic compound
TOC	total organic carbon
UAO	Unilateral Administrative Order
USEPA	U.S. Environmental Protection Agency

1 INTRODUCTION

This document presents the Field Sampling Plan (FSP) that has been prepared on behalf of International Paper Company (IPC) and McGinnes Industrial Maintenance Corporation (MIMC), for the 2010 soil study at the San Jacinto River Waste Pits (SJRWP) Superfund site (the Site). This FSP was prepared consistent with U.S. Environmental Protection Agency (USEPA) guidance (USEPA 1988, 1992) and as required by the USEPA 2009 Unilateral Administrative Order (UAO), Docket No. 06-03-10, which was issued by USEPA to IPC and MIMC on November 20, 2009 (USEPA 2009). Additional information on the Site history and a summary of existing data are provided in the Sampling and Analysis Plan (SAP). Information on geology, physiography, hydrology, and cultural and natural resources of the Site and information on fate and transport are provided in the Remedial Investigation and Feasibility Study (RI/FS) Work Plan (Anchor QEA and Integral 2010).

The Site consists of impoundments, built in the mid-1960s for disposal of paper mill wastes, and the surrounding areas containing sediments and soils potentially contaminated with the waste materials that had been disposed of in these impoundments. Two impoundments, together approximately 14 acres in size, are located on a 20-acre parcel immediately north of the I-10 bridge and on the western bank of the San Jacinto River, in Harris County, Texas (Figure A-1).

USEPA has identified an area south of I-10 to be investigated, based on historical documents and aerial photographs. These documents indicate that an additional impoundment was constructed south of I-10, on the peninsula of land directly south of the 20-acre parcel, and also was used as a paper mill waste disposal area in the mid-1960s for paper mill waste similar to that disposed of in the two impoundments (Figure A-1). A discussion of the impoundment south of I-10 and sampling to address related uncertainties is addressed in Soil SAP Addendum 1 and Soil FSP Addendum submitted on behalf of IPC.

The primary objective of the 2010 soil study is to collect information on chemical concentrations in soil at the Site. As discussed in the SAP, soil data will be used to delineate the nature and extent of contamination, support risk assessments (i.e., human health and

ecological), and to develop the conceptual site model and better understand potential contaminant fate and transport processes. Remedial actions and possible engineering alternatives will be conducted as a separate phase of the soil program as a part of the RI/FS.

To execute this study, Integral Consulting Inc. (Integral) and Anchor QEA, LLC (Anchor QEA) will conduct the fieldwork and data analysis for execution of Study Elements 1 through 3, as discussed in the SAP. The names and quality assurance (QA) responsibilities of key project personnel for Anchor QEA and Integral who will be involved in sampling and analysis activities are provided in Figure 1 of the SAP.

1.1 Overview

As a result of requirements relating to the time critical removal action (TCRA) underway a the Site, 31 of the total number (52) of soil sample locations on the Site and north of I-10 have already been sampled in three discrete sampling events as of December, 22 2010:

- August 2010: twelve stations in the TxDOT ROW, required for the TCRA
- October 2010: six stations within the northern impoundments, required for the RI/FS
- November 2010; thirteen stations on the upland sand separation area, north of I-10 and west of the northern impoundments

Sampling of three additional locations in Area 3 (surface and shallow subsurface samples at the locations of the groundwater well borings in the north impoundments) will be conducted as part of the groundwater sampling event during the final week of 2010. The remainder of sampling required by this SAP and addressing areas north of I-10 will therefore consist of 18 locations on the Site and north of I-10, and 20 locations in background areas. This work will be conducted as a discrete sampling event in January, 2011. Finally, Soil SAP Addendum 1 is in review by EPA, and addresses sample collection south of I-10, which will take place as an additional discrete sampling event. While all of the QA/QC, documentation and other requirements of this document and the main Soil SAP have been followed in these prior sampling events, the text below addresses the collection of samples that have not been sampled as of December 22, 2010.

The soil sampling design for the RI/FS incorporates a number of different components. The individual study components (as discussed in the SAP) differ in the locations and depths at

which soil is to be collected. Soil samples will be collected from the following areas (Figures A-2 and A-3):

- Area 1. The unvegetated portion of the upland area west of the impoundments and the road that provides access into and out of the upland area
- Area 2. The area underneath I-10
- Area 3. The area of the Site in which the impoundments north of I-10 occur
    - This includes surface and shallow subsurface soils at boring locations, as well as deep subsurface soils (5-foot intervals) to be collected from borings for groundwater well pairs.
- Area 4. The area in which waste handling south of I-10 may have affected soil (sampling in this area will be addressed in an addendum to this FSP).
- Background Areas. One or more of the background areas in the vicinity of the Site.

Soil samples will be analyzed for primary chemicals of potential concern (COPCs, several metals, bis[2-ethylhexyl]phthalate, and dioxins and furans), grain size, and total organic carbon (TOC). An archival sample jar will be collected for each sample for possible future analysis of secondary COPCs (polychlorinated biphenyls [PCBs], semivolatile organic compounds [SVOCs] and thallium). Soil samples not slated for analysis will be archived for possible future analysis of primary and/or secondary COPCs.

The sampling design elements are presented in Tables A-1 through A-4 and can be summarized as follows:

- **Area 1:** Surface and shallow subsurface soil sampling and analysis of COPCs at 18 locations from Area 1, in and near the upland area west of the impoundments and along the road extending to the west (stations SJTS014 through SJTS031; Figure A-2 and Table A-1), to support evaluation of the nature and extent of contamination, risk assessments, and contaminant fate and transport. Surface and shallow subsurface soil samples will be collected at all 18 stations at depths of 0 to 6 inches (0 to 15 cm) and 6 to 12 inches (15 to 30 cm). Deep subsurface samples from 12 to 24 inches (30 to 60 cm) will be collected at 8 of these stations (Figure A-2 and Table A-1). The analytical requirements for soil samples collected from Area 1 are as follows:

  - *Area 1 Surface and Shallow Subsurface Stations:* The surface and subsurface soil samples collected from depths of 0 to 6 inches (0 to 15 cm) and 6 to 12 inches (15 to 30 cm) from the 18 stations in Area 1 (Figure A-2) will be analyzed for primary COPCs, TOC, and grain size. An additional soil sample in a unique jar will be collected from each of these sample intervals for possible future analysis of secondary COPCs.
  - *Area 1 Deep Subsurface Stations:* The deep subsurface soil samples collected from 12 to 24 inches (30 to 60 cm) from the 8 stations in Area 1 (Figure A-2) will be analyzed for primary COPCs, TOC, and grain size. An additional soil sample in a unique jar will be collected from each of these sample intervals for possible future analysis of secondary COPCs.

- **Area 2.** Sampling was performed in August, 2010 (Anchor QEA 2010)
- **Area 3:** The surface and shallow subsurface soil samples collected from depths of 0 to 6 inches (0 to 15 cm) and 6 to 12 inches (15 to 30 cm) from the 9 soil sample stations in Area 3 (Figure A-2) will be analyzed for primary COPCs, TOC, and grain size. Stations for these samples correspond to the 3 locations for installation of groundwater well pairs (to be collected in late December, 2010) and 6 locations of geotechnical parameter measurements within the impoundments north of I-10 (collected in October, 2010). An additional soil jar will be collected from these sample intervals for possible future analysis of secondary COPCs. Sampling for the 6 locations followed the sample collection matrix provided in Table A-5. Samples from depths of 0 to 6 inches (0 to 15 cm) and 6 to 12 inches (15 to 30 cm) from the three locations corresponding to the groundwater well pairs will be collected as described here, and performed during execution of the Groundwater SAP.
- **Area 4.** Sampling will be described in an Addendum to this FSP.
- **Background Areas:** Surface and subsurface soil samples will be collected at 20 background stations from one or more areas shown on Figure A-3. The background soil sample locations will be determined on the basis of safety, accessibility, and permissions prior to initiating field activities. Soil samples will be collected at depths 0 to 6 inches (0 to 15 cm) and 6 to 12 inches (15 to 30 cm) and will be analyzed for primary COPCs, TOC, and grain size. An additional soil sample in a unique jar will be collected from these sample intervals for possible future analysis of secondary COPCs. Deep subsurface soil samples at 12 to 24 inches (30 to 60 cm) will not be

collected at the background area stations.

- **Soils from Groundwater Well Borings:** Deep subsurface soils samples will be collected during drilling of groundwater wells necessary for the groundwater study, and will be archived for possible future analysis of primary COPCs and TOC; samples will be analyzed for grain size and TOC (using the Walkley Black method, Table 8 of the main Soil SAP) immediately following collection. Samples collected from each 5-foot interval will be archived for possible future chemical analyses. Field personnel advancing the well borings will collect the soil samples according to the methods described in a standard operating procedure (SOP) that is attached to the Groundwater SAP (in progress); field forms and boring logs to be used for the soil samples from groundwater well borings are also provided in the Groundwater SAP.

1.2 Document Organization

This FSP describes the field methods that will be used to collect soil for components of the 2010 soil study addressing nature and extent and exposure assessment. The background, rationale, data quality objectives, and overall study design are described in detail in the SAP. Section 2 of this FSP describes the field procedures and sample packaging and shipping requirements that will be followed by the technical team during the field study. Section 3 summarizes field documentation and chain-of-custody (COC) procedures. Field data reporting and field custody procedures are discussed in Section 3.
The following documents are provided as attachments to this FSP:

- Attachment A1: Addendum 3 to the Overall Health and Safety Plan: Soil Sampling Health and Safety Plan. This document describes the specific requirements and procedures that will be implemented to minimize the safety risk to personnel who carry out the field study program for soil collection. It is an addendum to the project's overall health and safety plan (HASP; Anchor QEA 2009).
- Attachment A2: Standard Operating Procedures. The SOPs describe the procedures that will be used to collect surface and subsurface soils.
- Attachment A3: Field Forms. This attachment contains examples of various forms that will be used during field sampling, including a boring log, a field change request form, and a COC form.
- Attachment A4: USEPA Risk Assessment Guidance Forms (per the UAO Statement

of Work). This attachment contains the risk assessment guidance forms from USEPA (1992) that were stipulated in Item 17a of the UAO statement of work.

2 SAMPLING PROCEDURES

The following sections describe the detailed procedures and methods that will be used during the 2010 soil study, including sampling procedures, recordkeeping, sample handling, storage, and field quality control (QC) procedures. Sample collection and processing will be conducted in accordance with the SOPs provided in Attachment A2. Depending on field conditions, procedures specified in the referenced SOPs may be modified if necessary. All field activities will be conducted in accordance with the soil HASP addendum that is provided as Attachment A1.

2.1 Field Survey and Sampling Methods

The following sections present the soil sampling methodology.

2.1.1 Field Equipment and Supplies

Field equipment and supplies include sampling equipment, utensils, decontamination supplies, sample containers, coolers, shipping containers, log books and forms, personal protection equipment, and personal gear. Protective wear (e.g., nitrile gloves) is required to minimize the possibility of cross-contamination between sampling locations. Additional information on protective wear required for this project is provided in Attachment A1.

Surface soil samples 0 to 6 inches (0 to 15 cm) will be collected using stainless-steel shovels, trowels, or spoons. A coring device (e.g., hand-held corers, hand auger, or equivalent type of equipment) or stainless steel shovel will be used for subsurface soil sample collection.

Sample jars, preservatives, laboratory-grade distilled water, coolers, and packaging material for the samples will be supplied by the analytical laboratory. Details on the numbers and type of sample containers are provided in the SAP and in Table A-2 of this FSP. The field lead and field personnel in charge of sample handling in the field will use a sample matrix table (Table A-3) as a QC check to ensure that all samples have been collected at a given station and to record sample and tag numbers. This table includes the total number and type of sample jars required for each analysis at each sampling station. Table A-3 also includes the soil samples that will be collected during advancement of the monitoring well borings (Groundwater SAP, in progress), but these samples will be collected as part of groundwater

well installation, not at the same time as the nature and extent and exposure samples are collected. The Groundwater SAP and FSP (in progress) describe that sampling process.

Commercially available, pre-cleaned jars will be used for the samples, and the testing laboratories will maintain a record of certification from the suppliers. The bottle shipment documentation will include batch numbers. With this documentation, jars can be traced to the supplier, and bottle-wash analysis results can be reviewed. The bottle-wash certificate documentation will be archived in Integral's project file.

Sample containers will be clearly labeled at the time of sampling. Labels will include the task name, sample number, sampler's initials, analyses to be performed, and sample date and time. Sample numbering and identification procedures are described in detail in Section 3.5.

2.1.2 Sample Location Positioning

Latitude and longitude coordinates will be obtained at the locations where soil samples are collected. The station coordinates will be collected at a single location in the approximate middle of the subsample locations. A differential global positioning system (DGPS) will be used to document the sample collection locations. The standard projection method to be used during field activities is Horizontal Datum: NAD1983_StatePlane, Texas South Central, FIPS 4204, US feet. The positioning objective is to accurately determine and record the positions of all sampling locations to within ±2 m. Proposed soil sampling location coordinates are provided in Table A-4.

The DGPS unit consists of a global positioning system (GPS) receiver and a differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver. Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the appropriate quality of signal (SOP AP-06). The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoidance of these time intervals permits the operator to maintain better positioning accuracy (SOP AP-06).

2.1.3 Surface Soil Sample Collection

The equipment and procedures that will be used to collect surface soil samples during the 2010 soil study are discussed in the following sections. The estimated numbers of field locations that will be sampled are listed in Table A-1. The holding time requirements for the soil samples following field collection are specified in Table A-2. Soil samples will be collected in accordance with the sample matrix table (Table A-3).

Surface soil samples 0 to 6 inches (0 to 15 cm) may be collected with a variety of sampling equipment depending upon the conditions encountered in the field, including stainless-steel shovels, trowels, and spoons. At each of the sampling stations, a minimum of three grab samples will be collected within approximately 2 feet of each other. Any vegetative material or crushed concrete debris will be removed from the surface prior to sample collection and from the sample. A description of the type and depth of concrete/debris removed will be noted on the boring log or in the field logbook. Methods for surface soil sampling are provided in SOP SL-05 (Attachment A2).

One surface soil sample will be collected at each station location sampled for delineation of the nature and extent of contamination, to support exposure assessment, and to evaluate contaminant fate and transport. The samples will be analyzed for primary COPCs, TOC, and grain size. Additional soil from each sample will be archived for possible future analysis of secondary COPCs (Table A-3). The surface soil will be described in accordance with SOP SL-04 (Attachment A2) and American Society for Testing and Materials (ASTM) guidelines (ASTM 2000) on a boring log form (Attachment A3).

Surface soil will be placed into a decontaminated, stainless-steel bowl and homogenized using a stainless-steel spoon or other stainless-steel mixing implement until the soil attains a visually uniform color and texture. The soil sample in the bowl will be covered with aluminum foil until a sufficient quantity of soil (approximately 500 g per station) is collected. Soil subsamples will then be removed for the various kinds of laboratory analyses and for archiving.

The surface soil composite samples will be placed in labeled, laboratory-cleaned sample containers with Teflon-lined lids (Table A-2). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen at -20 degrees Celsius (i.e., archived samples) will have 0.5–1 inch (1.3–2.6 cm) of headspace above the soil to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice (4 ± 2°C).

As stated above, the sample matrix table (Table A-3) shows the total number of sample jars for each analysis needed at each sampling station. Integral's field lead and field personnel in charge of sample handling will use this table as a QC check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.1.4 Subsurface Soil Sample Collection

The equipment and procedures used to collect subsurface soil samples during the 2010 soil study are discussed in the following sections. The estimated numbers of field locations that will be sampled are listed in Table A-1. The holding time requirements for the soil samples following field collection are specified in Table A-2. Soil samples will be collected in accordance with the sample matrix table (Table A-3).

Subsurface soil samples from 6 to 12 inches (15 to 30 cm) will likely be collected with a stainless steel shovel. Soil samples from 12 to 24 inches (30 to 60 cm) will likely be collected using a hand auger through the middle of the holes dug to collect the 6 to 12 inch (15 to 30 cm) soil samples. Alternatively, a hand corer (or similar coring device) may be used to collect both subsurface soil samples. A minimum of three subsamples will be collected for the appropriate intervals within approximately 2 feet of each other at each station. If sample volume requirements dictate the need for additional soil, then additional subsamples will be collected and the soil will be composited from the appropriate interval. Any separate soil horizons that are observed in the core will be noted on the boring log (Attachment A3), but will not alter the collection interval provided in Table A-3.

Processing of the subsurface soil samples will occur in the field. The soil collected from each sample interval will be placed in a decontaminated stainless steel bowl and photographed. The photograph will include a tape measure for scale and a station identifier written on a white board. The subsurface soil samples will be described in accordance with SOP SL-04 (Attachment A2) and ASTM guidelines (ASTM 2000) on a boring log form (Attachment A3). Subsurface soil sample collection methods will generally follow the methods for surface soil sampling as specified in SOP SL-05 (Attachment A2).

The soil from each subsurface sample interval will be homogenized with a decontaminated stainless-steel mixing implement (e.g., spoon) until the soil attains a visually uniform color and texture. If a hand corer is utilized to collect the sample, soil touching the sides of the core tube will be excluded from each sample. The soil sample in the bowl will be covered with aluminum foil until a sufficient quantity of soil (approximately 500 g per station) is collected. Soil subsamples will then be removed for the various kinds of laboratory analyses and archiving.

The subsurface soil composite samples will be placed in labeled, laboratory-cleaned sample containers with Teflon-lined lids (Table A-2). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen (i.e., archived samples) will have 0.5 to 1 inch (1.3 to 2.6 cm) of headspace above the soil to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice (4 ± 2°C).

The subsurface soil samples will be archived or analyzed for primary COPCs, TOC, and grain size in accordance with Table A-3. Integral's field lead and field personnel in charge of sample handling in the field will use this table as a QC check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.1.5 Equipment Decontamination

Before sampling begins at a location, the sampling and compositing equipment (i.e., stainless-steel bowls, spoons, and hand auger) will be scrubbed with a standard detergent (e.g.,

Alconox® or Liquinox®), rinsed with water (potable, deionized, or distilled), and rinsed with laboratory-grade distilled or deionized water. After cleaning, the decontaminated sampling and compositing equipment will be covered with aluminum foil to protect it from possible contamination.

All non-dedicated sampling equipment that comes into contact with the soil samples (e.g., hand auger, core catchers, grab samplers, core liners, stainless-steel bowls, and utensils) will be decontaminated prior to use and between samples. Non-dedicated sampling equipment will be decontaminated following procedures in SOP SL-01 (Attachment A2), except that no solvent rinse will typically be used. If samples are collected that include obvious oily contamination, the sampling equipment used to collect and process them will be decontaminated using a separate decontamination station dedicated to heavily impacted equipment. This equipment will be wiped with a solvent following the initial decontamination, and it will undergo a second decontamination sequence using the standard decontamination procedures used for the non-oil-impacted equipment.

2.2 Field Quality Control Samples

Field QC samples will be used to assess sample variability and evaluate potential sources of contamination. The types of QC samples that will be collected for the 2010 soil study are described in this section. Detailed information on quality assurance and quality control (QA/QC) procedures, limits, and reporting are described in detail in the SAP. The estimated numbers of field QC samples to be collected are listed in the sample matrix table (Table A-3). If QC problems are encountered, they will be brought to the attention of Integral's QA coordinator. Corrective actions, if appropriate, will be implemented to meet the task's data quality indicators.

Field QC samples will include field split samples, standard reference materials, equipment filter wipe blanks, and filter blanks. The Field QC samples will be collected in accordance with SOP SL-02 (Attachment A2). The following QC samples will be collected in the field and analyzed by the analytical laboratory:

- Field split samples will be collected and analyzed to assess the variability associated with sample processing and laboratory variability. Blind field split samples will be

collected at a minimum frequency of 1 field split sample per 20 soil samples. A total of 5 field split samples will be collected during the soil study (Table A-3). Samples will be assigned unique numbers and will not be identified as field splits to the laboratory. Field split samples will be collected from both surface and subsurface soil samples for chemical analysis. A minimum of one field split sample will be collected for each kind of sample collected.

- Standard reference materials are samples of known concentration that have typically undergone multilaboratory analyses using a standard method. Reference materials provide a measure of analytical performance and/or analytical method bias. One standard reference material for soil will be submitted from the field and analyzed for dioxins and furans.
- Equipment filter wipe blanks will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., stainless-steel shovel, coring device, spoons, and mixing bowls). Equipment filter wipe blanks will be generated at approximately 5 percent of the soil sampling stations at a minimum, with at least one filter wipe blank collected for each type of sampling equipment. A total of 9 equipment filter wipe blanks will be collected during the soil study (Table A-3). One equipment filter wipe will be prepared for each analysis type. If multiple analyses are requested, separate sets of filter wipes will be collected for each analysis type for each kind of sampling equipment used, as the equipment can be wiped down only once for each piece of filter paper. This ensures that the filter wipe result represents the most conservative estimate of cross contamination for each analysis type. (Note: Filter papers must be stored in their original box, wrapped carefully in three layers of aluminum foil, or contained in a glass jar. The filter paper box cannot be stored in plastic bags or containers.). All equipment wipe samples will be clearly noted in the field log (e.g., sample identifier, equipment type, date and time of collection, analysis, and filter lot number).
- Filter blanks are prepared in the field to evaluate potential background concentrations present in filter paper used for the equipment filter wipe blank. Filter blanks will be collected at a minimum frequency of one for each lot number of filter papers used for collecting the equipment wipe blanks.

2.3 Sample Packaging and Transport

As mentioned above, sample coolers and packing materials will be supplied by the analytical laboratories. Sample packaging and transport will follow SOP AP-01 (Attachment A2). Individual sample jars will be labeled and placed into plastic bags and sealed. Samples will then be packed in a cooler lined with a large plastic bag. Glass jars will be packed to prevent breakage and separated in the cooler by bubble wrap or other shock-absorbent material. Ice in sealed plastic bags will then be placed in the cooler to maintain a temperature of approximately 4°C (±2°C). When the cooler is full, the COC form will be placed into a zip-locked bag and taped to the inside lid of the cooler. A temperature blank will be added to each cooler. Each cooler will be sealed with two COC seals, one each on the front and side of the cooler. Labels indicating "This End Up*"*with an arrow and "Fragile" will be attached to each cooler.

The shipping containers will be clearly labeled (i.e., name of task, time and date container was sealed, person sealing the cooler, and company name and address) for positive identification. These packaging and shipping procedures are in accordance with U.S. Department of Transportation regulations (49 CFR 173.6 and 49 CFR 173.24). Coolers containing samples for chemical analyses will be transported to the laboratory by courier or overnight shipping service.

After the chemistry samples have been received by the laboratory, they will be stored under refrigeration (4 ± 2°C). Archive soil samples collected from each composite sample for possible future analysis will be stored frozen at –20°C.

2.4 Study-Derived Wastes

Any excess phosphate-free, detergent-bearing liquid wastes from decontamination or any sample remaining after processing will be deposited in the vicinity of the collection area. Excess soils will be returned to the location where they were collected. Any dry waste (e.g., contaminated boots, bibs, Tyvek™ suits, contaminated soils) present at the end of the sampling event will be segregated and containerized (e.g., 55-gallon drums) and disposed of by a subcontractor specialized in hazardous waste removal. The subcontractor will be required to have, at a minimum, a drum management service that provides the following:

- Proper waste identification including full analytical capability
- Pickup and disposal of a broad range of hazardous wastes
- Safe and proper transportation
- Environmentally sound treatment and disposal
- Regularly scheduled service visits with manifest and label preparation.

All disposable materials used for sample collection and processing, such as paper towels and gloves, will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies that do not contain Site soil will be removed from the Site by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill.

3 FIELD DOCUMENTATION

The integrity of each sample from the time of collection to the point of data reporting must be maintained. Proper record-keeping and COC procedures will allow samples to be traced from collection to final disposition. Representative photographs will be taken of each area where samples are collected. A photograph will be taken of each surface and subsurface soil interval collected. Site photos from various angles and close-up views of the overall conditions will also be taken as necessary. Field documentation procedures will follow guidelines provided in SOP AP-02 (Attachment A2).

3.1 Field Log Book

All field activities and observations will be noted in a log book. The field log book will be a bound document and may contain individual field and sample log forms (depending on the sampling activity). Information will include personnel, date, time, station designation, sampler, types of samples collected, and general observations. Any changes that occur during sampling (e.g., personnel, responsibilities, or deviations from the FSP) and the reasons for these changes will be documented. The log book will identify on-site visitors (if any) and the number of photographs taken at each sampling location. Each field lead is responsible for ensuring that their respective field log book and all field data forms are correct. Requirements for log book entries will include the following:

- Log books will be bound, with consecutively numbered pages.
- Removal of any pages, even if illegible, will be prohibited.
- Entries will be made legibly with black (or dark) waterproof ink.
- Unbiased, accurate language will be used.
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be recorded, as well as the time of the observation itself).
- Each consecutive day's first entry will be made on a new, blank page.
- The date and time, based on a 24-hour clock (e.g., 0900 for 9:00 a.m. and 2100 for 9:00 p.m.), will appear on each page.

In addition to the preceding requirements, the person recording the information must initial and date each page of the field log book. If more than one individual makes entries on the

same page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry.

Log book corrections will be made by drawing a single line through the original entry, allowing the original entry to be read. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field log book and/or field data forms includes the following:

- Task name, task location, and task number
- Task start date and end date
- Weather conditions
- Name of person making entries and other field staff
- On-site visitors, if any
- Station number and location
- Date and collection time of each sample
- The sample number for each sample to be submitted for laboratory analysis
- The specific date and time with corresponding station number associated with the sampling location coordinates derived from DGPS
- Specific information on each type of sampling activity
- The sample number, date and time of collection, equipment type, and the lot number for the box of filter papers used for field QC samples
- Observations made during sample collection, including weather conditions, complications, and other details associated with the sampling effort
- Sample description (source and appearance, such as soil type, color, presence of anthropogenic material, and presence and type of biological structures, other debris, oil sheens, and odor)
- Soil penetration depth (nearest 0.5 cm) based on soil depth at the center of the excavation
- Any visible debris near any of the sampling locations
- Any surface vegetation that is removed from the sampling location prior to sampling
- The locations of any surface water runoff or seeps that are located near any of the sampling stations

- The number of photographs taken at the sampling location
- A record of Site health and safety meetings, updates, and related monitoring
- Any deviation from the FSP and reasons for deviation.

In addition, a sampling location map will be updated during sampling and will be maintained throughout the sampling event. All log books must be completed at the time that any observations are made. Copies of all log books and forms will be retained by the technical team.

3.2 Boring Logs

The field geologist will provide soil descriptions in accordance with SOP SL-06 (Attachment A2) and ATSM guidelines (ASTM 2000) for the soils on a standard boring log (Attachment A3). Boring logs will include the following information:

- Soil descriptions
- Date and time of collection of each soil sample
- Names of field personnel collecting and handling the samples
- Type of sampling equipment used (e.g., stainless steel, hand-corer)
- The sample station identification
- The sample number
- Length and depth intervals of each core section and estimated recovery (if applicable).

Samples to be collected from borings at groundwater wells will be recorded using boring logs specified in the related discussion in the Groundwater FSP.

3.3 Chain-of-Custody Procedures

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals (see SOP AP-03). A COC record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the COC will be included in laboratory and QA/QC reports. Attachment A3 contains an example of the COC form that will be used during the 2010 soil study.

At a minimum, the form will include the following information:

- Site name
- Field lead's name and team members responsible for collection of the listed samples
- Collection date and time for each sample
- Sample type (i.e., sample for immediate analysis or archive)
- Number of sample containers shipped
- Requested analyses
- Sample preservation information (if any)
- Name of the carrier relinquishing the samples to the transporter, noting date and time of transfer and the designated sample custodian at the receiving facility.

Integral's field lead will be the designated field sample custodian for their respective sampling events and will be responsible for all sample tracking and COC procedures for the samples that their respective teams collected in the field. The field sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The field sample custodian will complete COC forms prior to removing samples from the field. Upon transferring samples to the laboratory sample custodian (if a local laboratory is selected) or shipping courier (as appropriate), the field sample custodian will sign, date, and note the time of transfer on the COC form. The original COC form will be transported with the samples to the laboratories. All samples will be shipped to the testing laboratories in either coolers or shipping containers sealed with custody seals.

Each laboratory will designate a sample custodian who will be responsible for receiving samples and documenting their progress through the laboratory analytical process. The sample custodian for each laboratory will establish the integrity of the custody seals upon sample arrival at the laboratory. The laboratory sample custodian will also ensure that the COC and sample tracking forms are properly completed, signed, and initialed upon receipt of the samples.

When the laboratory receives the samples, the laboratory sample custodian will conduct an inventory by comparing sample labels to those on the COC document. The custodian will enter the sample number into a laboratory tracking system by task code and sample designation. The custodian will assign a unique laboratory number to each sample and will

be responsible for distributing the samples to the appropriate analyst or for storing samples at the correct temperature in an appropriate secure area.

3.4 Station Numbering

All stations will be assigned a unique identification code based on a designation scheme designed to suit the needs of the field personnel, data management, and data users. Soil sampling station numbers will include "SJ" to indicate San Jacinto followed by a two-letter code for the type of sample to be collected at a given location (TS). The letters will be followed by a three-digit number (e.g., 014, 020, 031). The station numbers will increase as the stations move to the west and south. An example soil station number for the 2010 soil study would be SJTS024.

Station numbers will not be recorded on sample labels or COC forms to prevent analytical laboratories from seeing the relationships between samples and stations.

3.5 Sample Identifiers

Samples will be labeled in accordance with SOP AP-04 (Attachment A2). Each soil sample from a given station will also have a unique label identifier. Sample identifiers will be established before field sampling begins and assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., field split samples) to ensure proper data analysis and interpretation (sample identification code); 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples (sample number); and 3) to track individual sample containers to ensure that the laboratory receives all of the material associated with a single sample (tag number). The sample identification code, sample number, and tag number are described below:

- A sample identification code for each surface sample will be created as follows: the station number (e.g., SJTS024), followed by an alpha character attached to the sample identifier that will distinguish between the different sample intervals (e.g., A = 0–6 inches [0–15 cm], B = 6–12 inches [15–30 cm], C = 12–24 inches [30–60 cm]). Example identifiers for a soil sample station would be SJTS024-A, SJTS024-B, and SJTS024-C. Field splits will include "-DUP" following the alpha character denoting

the sample interval. The sample identifier code will not be provided on the COC form since the codes contain station information and sample quality control information. Instead, the COC will contain a blind sample number to identify each sample (see next bullet).

- The sample number is an arbitrary number assigned to each soil sample collected (e.g., SL0001, SL0002) for chemical analysis. All subsamples of a composited field sample will have the same sample number. Each field split sample will have a different sample number, and the sample numbers of related field QC samples may not share any content. The sample number appears on the sample containers and the COC forms. Sample numbers will be assigned sequentially in the field.
- A unique numeric sample tag number will be attached to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container or will be shipped to multiple analytical laboratories, each container will have the same sample number and a different sample label with a unique sample tag number. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted). The sample tag number will appear on the COC forms. Tag numbers are used by laboratories only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number. Sample labels will be preprinted with tag numbers.

For equipment filter wipe blanks, sequential identifiers starting at 901 will be assigned instead of station numbers. For example, the first filter wipe blank for a surface soil sample collected with a stainless steel spoon and stainless steel bowl will be labeled as SSFW-901S, whereas the second filter wipe blank for a subsurface soil sample collected with a coring device will be labeled as SSFW-902C (SS = soil sample, FW = filter wipe, S = surface soil sampling equipment, and C = subsurface soil sampling equipment). The sample number for each equipment filter blank will be sequential beginning at FW0001.

4 FIELD DATA MANAGEMENT AND REPORTING PROCEDURES

During field operations, effective data management is critical to providing consistent, accurate, and defensible data and data products. Daily field records (a combination of field log books, field forms, if any, and COC forms) will make up the main documentation for field activities. Upon completion of sampling, field notes, data sheets (if any), and COC forms will be scanned to create an electronic record. Field data will be manually entered into the project database. One hundred percent of the transferred data will be verified based on hard copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

5 REFERENCES

Anchor QEA, 2009. Health and Safety Plan San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS.

Anchor QEA, 2010b. Draft TxDOT Right-of-Way Data Report. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS.

Anchor QEA and Integral, 2010. Revised Remedial Investigation/Feasibility Study Work Plan San Jacinto Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, Ocean Springs, MS, and Integral Consulting, Seattle, WA.

ASTM, 2000. Standard practice for description and identification of soils (Visual-Manual Procedure). Designation: D2488-00. American Society for Testing and Materials, West Conshohocken, PA.

USEPA, 1988. Interim Final Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 1992. Guidance for Data Usability in Risk Assessment. Parts A and B. Final. Publication 9285.7-09. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA, 2009. Unilateral Administrative Order for Remedial Investigation/Feasibility Study. U.S. EPA Region 6 CERCLA Docket No. 06-03-10. In the matter of: San Jacinto River Waste Pits Superfund Site Pasadena, Texas. International Paper Company, Inc. & McGinnes Industrial Management Corporation, respondents.

TABLES

Table A-1

Number of Soil Sampling Locations[a]

Sample Group	Sampling Method and Depth	Number of Locations[b]	Sample Locations	Analytes	Study Elements
Site surface soil	Stainless steel shovel, trowel, or spoon 0–6 inches (0–15 cm)	18	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment, fate and transport
Site subsurface soil	Stainless steel shovel, hand auger, or hand corer 6–12 inches (15–30 cm)	18	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site deep subsurface soil	Stainless steel shovel, hand auger, or hand corer 12–24 inches (30–60 cm)	8	Within Area 1	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Groundwater well boring soils	Well-boring equipment specified in the Groundwater SAP. All 5-foot intervals, composited across the depth of the sample increment.	3	Within Area 3	Lithology and grain size. Archival for chemistry.	CSM and fate and transport.
Groundwater well boring surface and shallow subsurface soils	Stainless steel shovel, hand auger, or hand corer 0–6 inches (0–15 cm), 6–12 inches (15–30 cm)	3	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site surface soil	Stainless steel shovel, trowel, or spoon 0–6 inches (0–15 cm)	6	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Site subsurface soil	Stainless steel shovel, hand auger, or hand corer 6–12 inches (15–30 cm)	6	Within Area 3	Primary COPCs, TOC, and grain size	Nature and extent, exposure assessment
Background surface soil	Stainless steel shovel, trowel, or spoon 0–6 inches (0–15 cm)	20	Background areas in the vicinity of the site	Primary COPCs, TOC, and grain size	Assessment of background conditions
Background shallow subsurface soil	Stainless steel shovel, hand auger, or hand corer 6–12 inches (15–30 cm)	20	Background areas in the vicinity of the site	Primary COPCs, TOC, and grain size	Assessment of background conditions

Notes

COPC = chemical of potential concern
TOC = total organic carbon

a - Additional samples will be collected south of I-10. The approach, design and sample numbers and purpose are described in Soil SAP Addendum 1.

b - Locations of samples collected for the TCRA are not included in these location counts.

Table A-2
Sample Containers, Preservation, and Holding Time Requirements

Matrix	Container [a] Type	Size	Laboratory	Parameter	Preservation	Holding Time	Sample Size [b]
Soil							
	WMG	8 oz.	TBD	TOC	4 ± 2°C	28 days	1 g
				Metals	4 ± 2°C	6 months	10 g
				Mercury	4 ± 2°C	28 days	5 g
	WMG	16 oz.	TBD	Grain size	4 ± 2°C	6 months	100 g
	WMG	8 oz.	TBD	Dioxins/furans	4 ± 2°C/Deep frozen (-20°C) [c]/ -10°C [d]	1 year/1 year [e]	50 g
	WMG	8 oz.	TBD	PCBs	4 ± 2°C/Deep frozen (-20°C) [c]/ -10°C [d]	1 year/1 year [e]	50 g
	WMG	8 oz.	TBD	SVOC/Archival	4 ± 2°C/ Deep frozen (-20°C) [c]	1 year [f]	50 g
Equipment Filter Wipe Blanks							
	HDPE	4 oz.	TBD	Metals	4 ± 2°C	6 months	1 wipe
	HDPE	4 oz.	TBD	Mercury	4 ± 2°C	28 days	2 wipe
	AG	4 oz.	TBD	Dioxins/furans	4 ± 2°C	1 year/1 year [e]	3 wipe
	AG	4 oz.	TBD	SVOCs	4 ± 2°C	7 days/40 days [e]	4 wipe

Notes

AG = amber glass
HDPE = high density polyethylene
PCB = polychlorinated biphenyl
SVOC = semivolatile organic compound
TBD = to be determined
TOC = total organic carbon
WMG = wide mouth glass
a - The size and number of containers may be modified by the analytical laboratory.
b - Sample sizes are estimated.
c - Samples will be shipped to the laboratory on ice at 4 ± 2°C. Once received at the laboratory, samples will be stored at -20°C.
d - Extracts will be stored at -10°C.
e - Holding time for samples prior to extraction/ holding time for extracts.
f - Holding time for frozen samples is 1 year.

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
Soil Sample Area 1															
☐ SJTS014	SJTS014-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS014-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS015	SJTS015-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS015-A-DUP	SL _ _ _ _	0-6 inches (0-15 cm)	Field Split	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS015-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS016	SJTS016-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS016-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS016-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS017	SJTS017-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS017-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS017-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS018	SJTS018-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS018-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS018-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ SJTS019	SJTS019-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS019-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS019-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS020	SJTS020-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS020-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS021	SJTS021-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS021-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS021-C	SL	12-24 inches (30-60 cm)	Normal	Tag #	Tag #	Tag #	Tag #	Tag #	Tag #	NA	NA	NA	NA	NA
☐ SJTS022	SJTS022-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS022-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS022-B-DUP	SL _ _ _ _	6-12 inches (15-30 cm)	Field Split	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS023	SJTS023-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS023-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS023-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS024	SJTS024-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS024-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS024-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ FW Blank	SSFW-901S	FW _ _ _ _	Surface Sampling Equipment	Equipment filter wipe blank[d]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ FW Blank	SSFW-902C	FW _ _ _ _	Shallow Subsurface Sampling Equipment	Equipment filter wipe blank[d]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ FW Blank	SSFW-903C	FW _ _ _ _	Deep Subsurface Sampling Equipment	Equipment filter wipe blank[d]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ Filter Paper	SSFB-904C	FB _ _ _ _	Filter paper	Filter blank[e]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ SJTS025	SJTS025-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS025-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS026	SJTS026-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS026-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS026-C	SL _ _ _ _	12-24 inches (30-60 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS026-C-DUP	SL _ _ _ _	12-24 inches (30-60 cm)	Field Split	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS027	SJTS027-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS027-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS028	SJTS028-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS028-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS029	SJTS029-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS029-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS030	SJTS030-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS030-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJTS031	SJTS031-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJTS031-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
Background Soil Sample Areas															
☐ SJBSS001	SJBSS001-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS001-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS002	SJBSS002-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS002-A-DUP	SL _ _ _ _	0-6 inches (0-15 cm)	Field Split	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS002-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS003	SJBSS003-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS003-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS004	SJBSS004-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS004-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS005	SJBSS005-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS005-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS006	SJBSS006-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS006-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS007	SJBSS007-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS007-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS008	SJBSS008-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS008-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS009	SJBSS009-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS009-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA

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Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ SJBSS010	SJBSS010-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS010-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ FW Blank	SSFW-904S	FW _ _ _ _	Surface Sampling Equipment	Equipment filter wipe blank[d]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ SJBSS011	SJBSS011-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS011-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS012	SJBSS012-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS012-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS012-A-DUP	SL _ _ _ _	6-12 inches (15-30 cm)	Field Split	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS013	SJBSS013-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS013-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS014	SJBSS014-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS014-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS015	SJBSS015-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS015-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS016	SJBSS016-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS016-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS017	SJBSS017-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS017-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS018	SJBSS018-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS018-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ FW Blank	SSFW-905C	FW _ _ _ _	Subsurface Sampling Equipment	Equipment filter wipe blank[d]	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ SJBSS019	SJBSS019-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS019-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
☐ SJBSS020	SJBSS020-A	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
	SJBSS020-B	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA	NA	NA
Groundwater Monitoring Well Borings															
☐ SJMWS01[f]	SJMWS01-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS01-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS01-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS01-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS01-0-6	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS01-6-12	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
☐ SJMWD01[f]	SJMWD01-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD01-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD01-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD01-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD01-E	SL _ _ _ _	20-25 feet (6-7.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD01-F	SL _ _ _ _	25-30 feet (7.5-9 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ SJMWS02[f]	SJMWS02-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS02-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS02-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS02-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS02-0-6	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS02-6-12	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
☐ SJMWD02[f]	SJMWD02-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD02-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD02-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD02-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD02-E	SL _ _ _ _	20-25 feet (6-7.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD02-F	SL _ _ _ _	25-30 feet (7.5-9 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA

Table A-3
Field Sample Collection Matrix

Station Number	Sample Identifier	Sample Number	Sample Depth	Sample Type	Soil Sample Analyses						Blank Filter Wipes				
					Primary				Secondary		Ghost Wipe		Whatman Grade 42 Filters		
					TOC, Metals (including mercury)	Grain Size	BEHP	Dioxins and Furans	Archive: PCBs	Archival	Metals	Mercury	BEHP	Dioxins and Furans	SVOCs
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C)[b]/-10 °C	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C/ Deep frozen (-20°C)	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ SJMWS03[f]	SJMWS03-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS03-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS03-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS03-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS03-0-6	SL _ _ _ _	0-6 inches (0-15 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWS03-6-12	SL _ _ _ _	6-12 inches (15-30 cm)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
☐ SJMWD03[f]	SJMWD03-A	SL _ _ _ _	0-5 feet (0-1.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD03-B	SL _ _ _ _	5-10 feet (1.5-3 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD03-C	SL _ _ _ _	10-15 feet (3-4.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD03-D	SL _ _ _ _	15-20 feet (4.5-6 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD03-E	SL _ _ _ _	20-25 feet (6-7.5 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA
	SJMWD03-F	SL _ _ _ _	25-30 feet (7.5-9 m)	Normal	NA	Tag #_____	NA	NA	NA	Tag #_____	NA	NA	NA	NA	NA

Definitions

NA = not applicable
PCB = polychlorinated biphenyl
SVOC = semivolatile organic compound
WMG = wide mouth glass

a - The size and number of containers may be modified by the analytical laboratory.

b - A unique numeric sample tag number will be attached to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, each container will have the same sample number and a different sample label with a unique sample tag number. A sample will also be split between containers if a different preservation technique is used for each container (e.g., freezing archive sample). The sample tag number will appear on the COC forms. Tag numbers are used by laboratories only to confirm that they have received all of the containers that were filled and shipped. Date will be reported by sample number.

c - Blind field split samples will be collected at a minimum frequency of 1 field split sample per 20 sediment samples.

d - A filter wipe blank sample will be collected at a minimum frequency of 1 per 20 sediment samples. One equipment wipe will be prepared for each analysis type. Because multiple analyses types are requested for this study, separate tests of filter wipes will be collected for each analysis type for each kind of sampling equipment used, as the equipment can be wiped down only once with each piece of filter paper. This ensures that the filter wipe result represents the most conservative estimate of cross contamination for each analysis type.

e - Filter blanks are prepared in the field to evaluate potential background concentration present in filter paper used for the equipment filter wipe blank. Filter blanks will be collected at a minimum frequency of one for each lot number of filter papers used for collecting the equipment wipe blank. The filter lot number will be clearly noted in the field logbook.

f - Composite soil samples will be collected at 5 foot intervals to the bottom of the well borings. The total depth of the well borings will be determined in the field.

Table A-4
Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Intervals	Analysis	Coordinates[a]	
				X	Y
SJTS014	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215913.408	13858827.64
SJTS015	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215843.408	13858687.64
SJTS016	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215983.408	13858687.64
SJTS017	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215913.408	13858547.64
SJTS018	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3216116.499	13858537.56
SJTS019	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215973.851	13858422.19
SJTS020	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216092.133	13858413.57
SJTS021	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215838.125	13858355.71
SJTS022	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215213.408	13858827.64
SJTS023	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215143.408	13858687.64
SJTS024	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215283.408	13858687.64
SJTS025	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215073.408	13858547.64

Table A-4
Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Intervals	Analysis	Coordinates[a]	
				X	Y
SJTS026	Surface, subsurface, and deep subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches), 30-60 cm (12-24 inches)	Primary COPCs, TOC, grain size	3215213.408	13858547.64
SJTS027	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215047.916	13858441.49
SJTS028	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215067.46	13858114.82
SJTS029	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3215027.006	13857909.68
SJTS030	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3214636.681	13857882.71
SJTS031	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3214209.124	13857756.77
SJGB001	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216697.5	13857564.5
SJGB006	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3217009.085	13857746.5
SJGB009	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216878.366	13857329.24
SJGB010	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216757.058	13857434.53
SJGB011	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216934.356	13857529.67
SJGB012	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216840.356	13857619.17
SJMWS01	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3216663	13857373
SJMWS02	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3217053	13857791

Table A-4
Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Intervals	Analysis	Coordinates[a]	
				X	Y
SJMWS03	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	3217206	13857112
SJBSS001	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS002	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS003	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS004	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS005	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS006	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS007	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS008	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS009	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS010	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS011	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS012	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS013	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD

Table A-4
Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Intervals	Analysis	Coordinates[a]	
				X	Y
SJBSS014	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS015	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS016	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS017	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS018	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS019	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD
SJBSS020	Surface and subsurface soil	0-15 cm (0-6 inches), 15-30 cm (6-12 inches)	Primary COPCs, TOC, grain size	TBD	TBD

Notes
COPC = chemical of potential concern
TBD = to be determined
TOC = total organic carbon

a - NAD 1983; State Plane Texas South Central FIPS 4204; US feet

Table A-5
Field Sample Collection Matrix for Area 3 (collected October 2010)

					Supplemental Chemistry Samples					Blank Filter Wipes			
					Primary				Archive	Ghost Wipes		Whatman Grade 42 filters	
Station	Sample ID	Sample Type	Sample Depth	Sample Group	TOC, Metals, Mercury, and Percent Moisture (EPA 160.3)	Grain Size	BEHP	Dioxins/ Furans		Metals	Mercury	BEHP	Dioxins/ Furans
					8 oz WMG[a]	16 oz WMG[a]	8 oz WMG[a]	8 oz WMG[a]	16 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]	4 oz WMG[a]
					4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C/ Deep frozen (-20°C) at lab	4 ± 2°C	4 ± 2°C	4 ± 2°C	4 ± 2°C
☐ SJGB001	SJGB001-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _[b]	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB001-CR1-B	Stainless-steel shovel	6–12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
☐ SJGB006	SJGB006-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB006-CR1-B	Stainless-steel shovel	6–12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
☐ SJGB009	SJGB009-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB009-CR1-B	Stainless-steel shovel	6–12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB009-CR1-B-DUP	Stainless-steel shovel	6–12 inches (15 -30 cm)	Field Split[c]	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
☐ SJGB010	SJGB010-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB010-CR1-B	Stainless-steel shovel	6 - 12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
☐ SJGB011	SJGB011-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB011-CR1-B	Stainless-steel shovel	6–12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
☐ FW Blank	GBFW-912C	Stainless-steel shovel	Stainless-steel shovel blade	Equipment filter wipe blank[d]						TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _
☐ Filter Paper	GBFB-913C	NA	Filter paper	Filter blank[e]						TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _
☐ SJGB012	SJGB012-CR1-A	Stainless-steel shovel	0–6 inches (0–15 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				
	SJGB012-CR1-B	Stainless-steel shovel	6–12 inches (15–30 cm)	Source station	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _	TG_ _ _ _				

[a] The size and number of containers may be modified by the analytical laboratory.

[b] A unique numeric sample tag number will be attached to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, each container will have the same sample number and a different sample label with a unique sample tag number. A sample will also be split between container if a different preservation technique is used for each container (e.g., freezing archive sample). The sample tag number will appear on the COC forms. Tag numbers are used by laboratories only to confirm that they have received all of the containers that were filled an shipped. Date will be reported by sample number.

[c] Blind field split samples will be collected at a minimum frequency of 1 field split sample per 20 sediment samples.

[d] A filter wipe blank sample will be collected at a minimum frequency of 1 per 20 sediment samples. One equipment wipe will be prepared for each analysis type. Because multiple analyses types are requested for this study, then separate tests of filter wipes will be collected for each analysis type for each kind of sampling equipment used, as the equipment can be wiped down only once with each piece of filter paper. This ensures that the filter wipe result represents the most conservative estimate of cross contamination for each analysis type.

[e] Filter blanks are prepared in the field to evaluate potential background concentration present in filter paper used for the equipment filter wipe blank. Filter blanks will be collected at a minimum frequency of one for each lot number of filter papers used for collecting the equipment wipe blank. The filter lot number will be clearly noted in the field logbook.

FIGURES

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ANCHOR QEA

integral consulting inc.

0 800

Scale in Feet

Mean High Water (+1 ft NAVD88)

USEPA's Preliminary Site Perimeter

Original (1966) Perimeter of the Northern Impoundments

Area of Soil Investigation South of I-10

[a] Designation of the sand separation area is intended to be a general reference to areas in which such activities are believed to have taken place based on visual observations of aerial photography from 1998 through 2002.

FEATURE SOURCES:
Aerial Imagery: 0.5-meter. Photo Date: 01/14/2009
Texas Strategic Mapping Program (StratMap), TNRIS

Figure A-1
Overview of Soil Study Area
SJRWP Soil FSP
SJRWP Superfund/MIMC and IPC

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ANCHOR QEA

integral consulting inc.

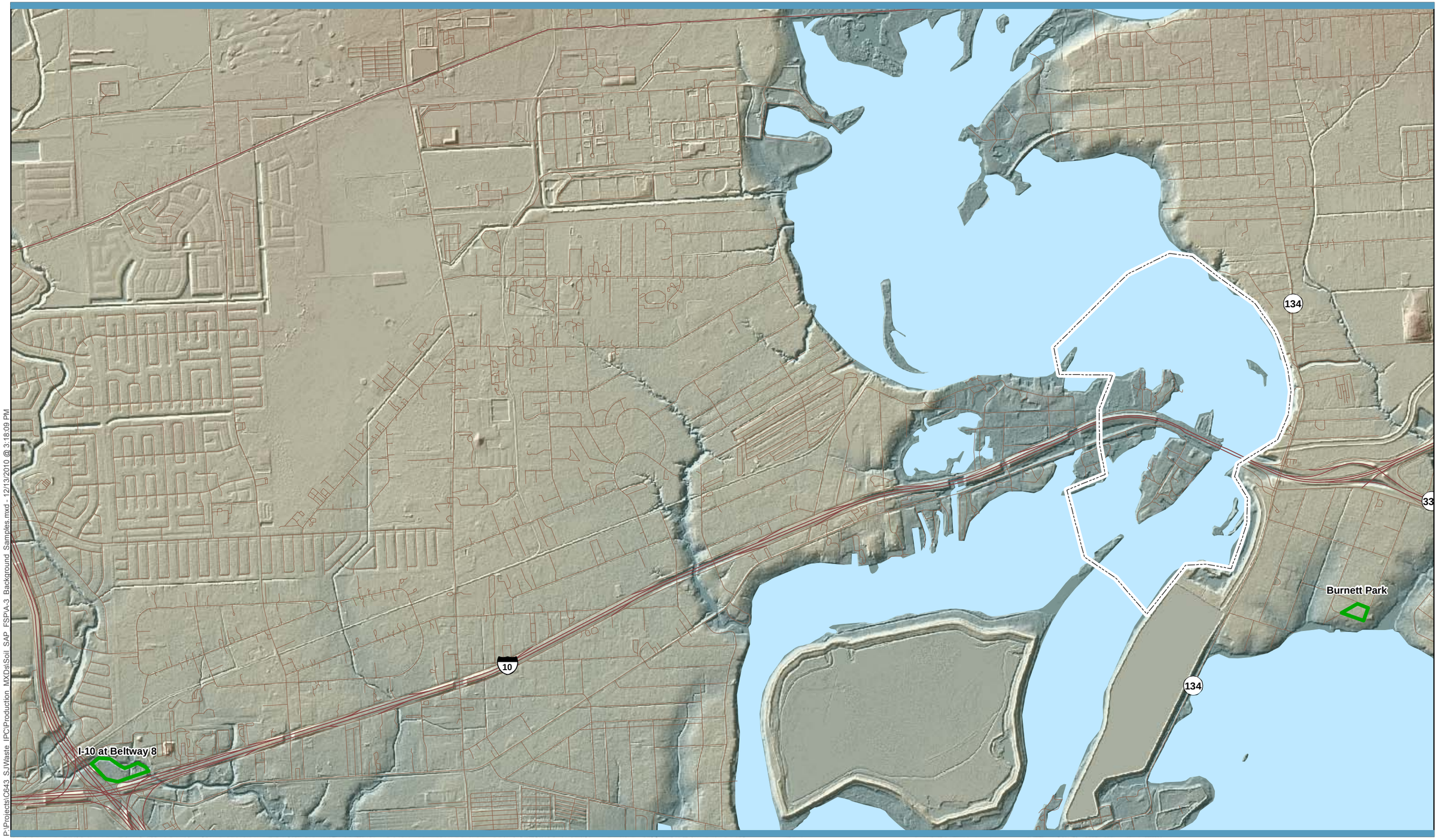
0 400

Scale in Feet

- Groundwater well/soil sampling station
- TCRA Soil
- TxDOT ROW Station
- RI/FS Surface Sediment Station
- USEPA's Preliminary Site Perimeter
- Soil Sampling Location (0-6 and 6-12 in)
- Soil Sampling Location (12-24 in)
- Proposed Contractor Access and Laydown Area
- MHW (+1 ft NAVD88)

FEATURE SOURCES:
Aerial Imagery: 0.5-meter 2008/2009 DOQQs-
Texas Strategic Mapping Program (StratMap)

Figure A-2
Soil Sample Locations
for the Area North of I-10
SJRWP Soil FSP
SJRWP Superfund/MIMC and IPC



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ANCHOR QEA integral consulting inc.

0 4,000

Scale in Feet

USEPA's Preliminary Site Perimeter

County Park

Figure A-3

Background Soil Sampling Locations

SJRWP Soil FSP

SJRWP Superfund/MIMC and IPC

ATTACHMENT A1
ADDENDUM 3 TO THE OVERALL HEALTH AND SAFETY PLAN:
SOIL SAMPLING HEALTH AND SAFETY PLAN

Prepared for

McGinnes Industrial Maintenance Corporation

International Paper Company

Prepared by

Integral Consulting Inc.

411 First Avenue South, Suite 550

Seattle, Washington 98104

December 2010

CERTIFICATION PAGE

Addendum 3 to the overall health and safety plan (HASP; Anchor QEA 2009) for the San Jacinto River Waste Pits Superfund Site (the Site) has been reviewed and approved by Integral Consulting Inc. (Integral) for the 2011 soil study at the Site in support of the remedial investigation and feasibility study (RI/FS) for the Site.

Jennifer Sampson	Bill Lawrence
Project Manager	Field Lead
Integral Consulting Inc.	Integral Consulting Inc.
Date: _____	Date: _____

HEALTH AND SAFETY PLAN ACKNOWLEDGEMENT FORM

Project Name: San Jacinto River Waste Pits Superfund Site

Addendum 3 to the overall HASP (Anchor QEA 2009) is approved by Integral for use at the San Jacinto River Waste Pits Superfund Site (the Site). The overall HASP and Addendum 3 are the minimum health and safety standard for the Site and will be strictly enforced for Integral personnel and other consulting personnel including subcontractors where applicable.

I have reviewed Addendum 3, dated December 2010, to the overall HASP for the 2011 soil study. I have had an opportunity to ask any questions I may have and have been provided with satisfactory responses. I understand the purpose of the plan, and I consent to adhere to its policies, procedures, and guidelines while an employee of Integral, or its subcontractors.

Date	Name (print)	Signature	Company

Date	Name (print)	Signature	Company

SITE EMERGENCY PROCEDURES

Emergency Contact Information

Table A

Site Emergency Form and Emergency Phone Numbers

Category	Information	
Chemicals of Potential Concern	Dioxins/Furans, aluminum, magnesium, mercury, and copper	
Minimum Level of Protection	Level D	
Site(s) Location Address	(No formal address, see Figure A) Channelview, TX 77530 Coordinates [29° 47' 38.49"N, 95° 3' 49.55"W]	
Emergency Phone Numbers		
Ambulance	911	
Fire	911	
Police	911	
Poison Control	911 and then 1-800-222-1212 if appropriate	
Project-Specific Health and Safety Officers' Phone Numbers		
Integral Field Lead (FL) and Integral Site Safety Officer (SSO)	Bill Lawrence	Office: (206) 957-0326 Cell: (206) 691-2216
Integral Corporate Health and Safety Manager (CHSM)	Eron Dodak	Office: (503) 284-5545 ext. 14 Cell: (503) 407-2933
Integral Project Manager (PM)	Jennifer Sampson	Office: (206) 957-0351 Cell: (360) 286-7552
Anchor QEA PM	David Keith	Office: (228) 818-9626 Cell: (228) 224-2983
Anchor QEA FL and SSO	Chris Torell	Office: (315) 453-9009 ext. 17 Cell: (315) 254-4954
Anchor QEA CHSM	David Templeton	Office: (206) 287-9130 Cell: (206) 910-4279
Client Contact – McGinnes Industrial Maintenance Corporation (MIMC)	Andrew Shafer	Office: (713) 772-9100 ext. 109 Cell: (832) 724-3802
Client Contract – International Paper Company (IPC)	Phil Slowiak	Office: (901) 419-3845 Cell: (901) 214-9550
Reporting Oil and Chemical Spills		
National Response Center	1-800-424-8802	
State Emergency Response System	(512) 424-2138	
EPA Environmental Response Team	(201) 321-6600	

Note: In the event of any emergency, contact both the Integral and Anchor QEA PMs and FLs.

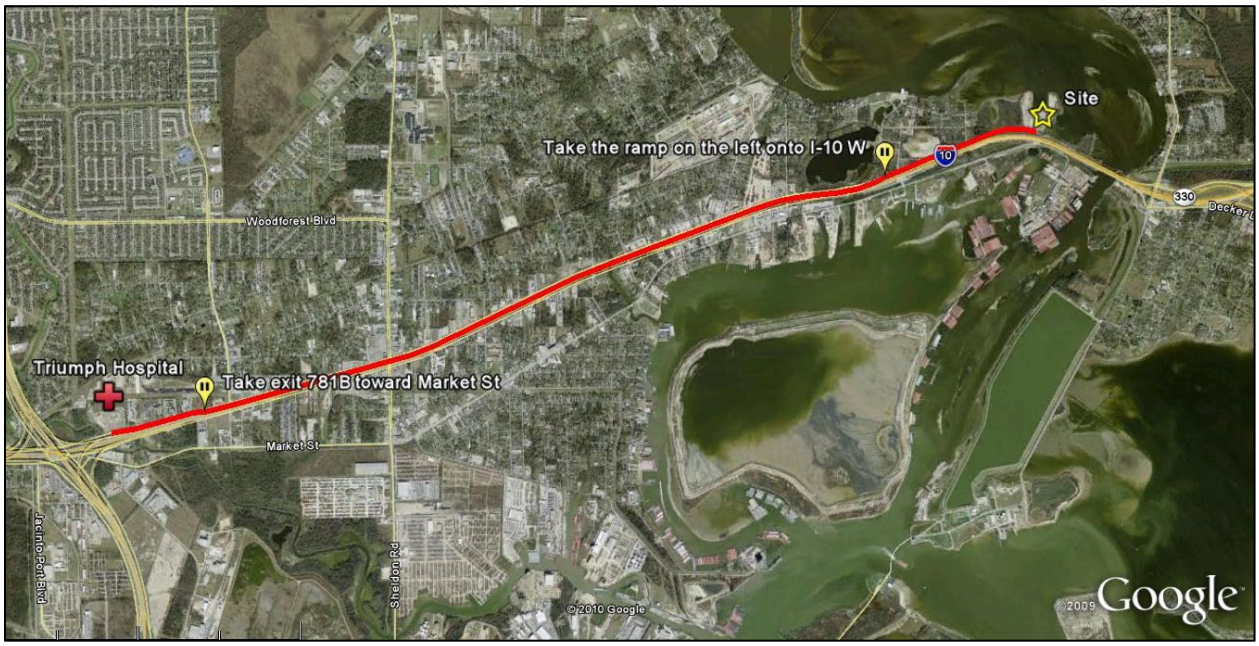
Figure A
Site Location Map

Table B
Hospital Information

Category	Information
Hospital Name	Triumph Hospital – East Houston
Address	15101 East Freeway
City, State	Channelview, TX 77530-41041
Phone	(713) 691-6556
Emergency Phone	(713) 691-6556

Figure B

Hospital Route Map



DRIVING DIRECTIONS FROM SITE TO HOSPITAL

1. Head west on East Freeway Service Road toward Monmouth Street (approximately 0.9 mile).
2. Take the ramp on the left to I-10 West.
3. Proceed on I-10 West to Exit 781B (approximately 3.7 miles).
4. Exit freeway at Exit 781B onto East Freeway Service Road.
5. Continue heading west on East Freeway Service Road (approximately 0.2 mile).
6. Triumph Hospital will be on the right (total distance approximately 5 miles).

Figure C

Hospital Detail (Egress from I-10 West)

Emergency Response Procedures

In the event of an emergency, refer to the procedures in the San Jacinto River Waste Pits Superfund Site Overall HASP (Anchor QEA 2009).

A copy of this Addendum must be included with the overall HASP, and both copies must be available in the field at all times during fieldwork.

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LIST OF ACRONYMS AND ABBREVIATIONS

Abbreviation	Definition
°F	degrees Fahrenheit
ACGIH	American Conference of Governmental Industrial Hygienists
Anchor QEA	Anchor QEA, LLC
AWG	American Wire Gauge
CHSM	Corporate Health and Safety Manager
COPC	chemical of potential concern
FL	Field Lead
FSP	Field Sampling Plan
HASP	Health and Safety Plan
Integral	Integral Consulting Inc.
IPC	International Paper Company
JHA	Job Hazard Analysis
MIMC	McGinnes Industrial Maintenance Corporation
mg/m^3	milligrams per cubic meter
MSDS	Material Safety Data Sheets
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Act or Administration
PEL	Permissible Exposure Limit
PM	Project Manager
PPE	personal protective equipment
RI/FS	remedial investigation and feasibility study
Site	San Jacinto River Waste Pits Superfund Site
SJRWP	San Jacinto River Waste Pits
SSO	Site Safety Officer
STEL	Short Term Exposure Limit
TLV	Threshold Limit Values
TOC	total organic carbon
TWA	Time Weighted Average
USEPA	U.S. Environmental Protection Agency

1 INTRODUCTION

Integral Consulting Inc. (Integral) has prepared Addendum 3 to the San Jacinto River Waste Pits (SJRWP) Superfund Site (the Site) overall Health and Safety Plan (HASP; Anchor QEA 2009). This addendum provides study-specific information and health and safety provisions to protect workers from potential hazards during soil sampling activities at locations in the Site and within the San Jacinto River. Site background information and general health and safety provisions to protect workers from potential hazards during work at the Site are presented in the overall HASP.

The provisions of this Soil Sampling HASP are mandatory for all Integral, Anchor QEA, LLC (Anchor QEA), and any contractor personnel assigned to the project. Other contractors that will be working at the Site are also expected to follow the provisions of this Soil Sampling HASP unless they have their own HASP that covers their specific activities related to this study and such HASPs have been approved by Integral. Any other contractor HASPs must include the requirements set forth in this Soil Sampling HASP and the overall HASP (Anchor QEA 2009), at a minimum. All visitors to the work Site, including U.S. Environmental Protection Agency (USEPA) personnel; state and local government personnel; or employees, representatives, or contractors of McGinnes Industrial Maintenance Corporation (MIMC) and International Paper Company (IPC) must also abide by the requirements of this Soil Sampling HASP and will attend a pre-work briefing where the contents of this Soil Sampling HASP and the overall HASP (Anchor QEA 2009) will be presented and discussed.

It is Integral's policy to provide a safe and healthful work environment. No aspect of the work is more important than protecting the health and safety of all workers.

Integral cannot guarantee the health or safety of any person entering the Site. Because of the potentially hazardous nature of the Site and the activity occurring thereon, it is not possible to regulate personal diligence or to discover, evaluate, and provide protection for all possible hazards that may be encountered. Strict adherence to the health and safety guidelines set forth herein and in the overall HASP (Anchor QEA 2009) will reduce, but not eliminate, the potential for injury and illness at the Site. The health and safety guidelines in this plan were

prepared specifically for the Site and should not be used on any other site without prior evaluation by trained health and safety personnel.

A copy of this Soil Sampling HASP (Addendum 3) and the overall HASP (Anchor QEA 2009) must be in the custody of the field crew during field activities. All individuals performing field work must read, understand, and comply with these plans before undertaking field activities. Once the information has been read and understood, the individual must sign the Site Health and Safety Acknowledgment Form provided with this Soil Sampling HASP. The signed form will become part of Integral and Anchor QEA project files (as applicable to each company).

Addendum 3, this Soil Sampling HASP, may be modified at any time based on the judgment of either Integral's or Anchor QEA's Site Safety Officer (SSO) in consultation with Integral's or Anchor QEA's Corporate Health and Safety Manager (CHSM) and Project Manager (PM) or designee. Any modification will be presented to the on-site team during a safety briefing and will be recorded in the field notebook.

2 SCOPE OF WORK

The soil sampling design for the remedial investigation and feasibility study (RI/FS) incorporates a number of different components. The individual study components (as discussed in the Sampling and Analysis Plan) differ in the locations and depths at which soil is to be collected. Soil samples will be collected from the following areas (see Figures A-2 and A-3 of the Field Sampling Plan [FSP]):

- Area 1. The unvegetated portion of the upland area west of the impoundments, including the area surrounding the road that provides access in and out of the upland area
- Area 2. Already sampled.
- Area 3. The vegetated portion on the western side of the impoundments on the north side of I-10.
- Area 4. The upland area of the peninsula south of I-10 (discussed in the Soil SAP Addendum); a HASP Addendum specific to Area 4 is included in the Soil SAP FSP Addendum.
- Background Areas. One or more of the background areas in the vicinity of the Site.

The sampling design can be summarized as follows:

- **Area 1:** Surface and subsurface soil sampling and analysis of chemicals of potential concern (COPCs) at 18 locations from Area 1, in and near the uplands west of the impoundments and along the road extending to the west (stations SJTS014 through SJTS031; Figure A-2 of the FSP), to support evaluation of nature and extent of contamination, risk assessments, and development of the conceptual site model. Surface and subsurface soil samples will be collected at all 18 stations at depths of 0 to 6 inch (0 to 15 cm) and 6 to 12 inches (15 to 30 cm); samples of deep subsurface soil from 12 to 24 inches (30 to 60 cm) will be collected from 8 of the 18 locations; all samples will be analyzed for primary COPCs, total organic carbon (TOC), and grain size.
- **Area 3:** Already sampled.
- **Area 4:** Details of the sampling activities will be included in the HASP Addendum of the Soil SAP FSP Addendum.

- **Background Areas:** Surface and subsurface soil samples will be collected at 20 background stations from one or more areas shown on Figure A-3 of the FSP. The background soil sample locations will be determined on the basis of safety, accessibility, and permissions prior to initiating field activities. Soil samples will be collected at depths of 0 to 6 inch (0 to 15 cm) and 6 to 12 inches (15 to 30 cm) and will be analyzed for primary COPCs, TOC, and grain size. An additional soil jar will be collected from these sample intervals for possible future analysis of secondary COPCs. Deeper subsurface soil samples will not be collected at the background area stations.

The surface and subsurface soil samples will be collected using a stainless-steel shovel, trowel, spoon, hand auger, or hand corer.

3 AUTHORITY AND RESPONSIBILITIES OF KEY PERSONNEL

This section describes the authority and responsibilities of key Integral and Anchor QEA project personnel.

To maintain adequate Site control, the SSO will have the authority to enforce the rules of the overall HASP and the Soil Sampling HASP Addendum 3 to any individual present at the Site, whether that individual is an employee or an outside contractor who is working with his or her team.

Because there is more than one HASP (i.e., overall HASP [Anchor QEA 2009] and the Soil Sampling HASP [Addendum 3]), the Occupational Safety and Health Act or Administration (OSHA) (OSHA 1997) considers it essential that the plans be integrated and enforced consistently to ensure that on-site personnel have a clear understanding of health and safety expectations, lines of authority, and emergency response actions.

The names and contact information for key safety personnel are listed in the Emergency Site Procedures section at the beginning of this HASP (Table A). Should key Site personnel change during the course of the project, a new list will be established and given immediately to the field team. The emergency phone number for the Site is **911**, and should be used for all medical, fire, and police emergencies.

Eron Dodak (proposed Field Lead [FL] and SSO) has oversight responsibility for all safety and health activities and the authority to discontinue or modify Site operations when unsafe conditions are detected. Eron Dodak is also Integral's CHSM and will be in direct contact with Integral's PM (Jennifer Sampson) during the fieldwork.

The PM will be in regular contact with the FL/SSO/CHSM to ensure that appropriate health and safety procedures are implemented during the 2011 soil study.

4 JOB HAZARD ANALYSIS

The OSHA standard (29 CFR 1910.120) mandates that Site safety and health programs require that task- and operation-specific hazard analyses be conducted at the Site. These analyses are intended to ensure a comprehensive and systematic approach to hazard anticipation, recognition, and evaluation at hazardous waste sites.

The kinds of potential hazards associated with soil sampling are summarized in the Job Hazard Analysis (JHA) that is provided in Table 1 (located at the end of this section) for the soil sampling task. The JHA lists a task or operation required during Site activity and the location(s) where that task or operation is performed. A single JHA may be used for a task performed in multiple locations if the hazards, potential exposures, and controls are the same in each location.

The JHA lists the chemical hazards associated with that task and their known or anticipated airborne concentrations during performance of the task. Each JHA also identifies anticipated physical and biological hazards and potential exposure levels or the likelihood of exposure. The final section of each JHA lists the control measures implemented to protect employees from exposure to the identified hazards. The information provided here is designed to satisfy OSHA's hazardous waste operations and emergency response JHA requirements of 1910.120(b)(4)(ii)(A) and the workplace hazard assessment requirements of 1910.132(d).

Health hazard information for all COPCs identified in Site JHAs appears in the material safety data sheets (MSDSs) of the overall HASP (Anchor QEA 2009).

Integral's FL will modify the study-specific JHA when:

- The scope of work is changed by adding, eliminating, or modifying tasks
- New methods of performing study tasks are selected
- Observation of the performance of study tasks results in a revised characterization of the hazards
- New chemical, biological, or physical hazards are identified
- Exposure data indicate changes in the concentration and/or likelihood of exposure

New/different control measures are selected.

Table 1

Job Hazard Analysis for Soil Sampling – Types of Potential Hazards

Operational Phase: SJRWP RI/FS	Location: Areas 1 through 3 at the SJRWP; background areas near the SJRWP.

Chemical Hazards

Chemical of Potential Concern	PEL - TWA[1] mg/m^3	TLV - TWA[2] mg/m^3	STEL mg/m^3	Ceiling Limit mg/m^3	Exposure Routes	Symptoms
2,3,7,8-TCDD	-	-	-	-	Inh, Abs, Ing, Con	Irritation to eyes, chloracne, dermatitis
Mercury	0.01	0.025	0	0.1	Inh, Ing, Con	Irritation to eyes, skin, cough, chest pain, dyspnea, bronchitis, pneumonitis; tremor, insomnia, irritability, indecision, headache, lassitude; stomatitis, salivation; gastrointestinal disturbance, anorexia, weight loss; proteinuria
Copper (fume)	0.1	0.2	-	-	Inh, Con	Fever, chills, muscle ache, nausea, dry throat, weakness

Physical Hazards

Name of Physical Hazard	Source	Exposure Level/ Potential	Exposure Limit
Boating operations	N/A	N/A	N/A
Pinch and crush zones	N/A	N/A	N/A
Drowning	N/A	N/A	N/A
Heat (ambient)	Sun	Likely	N/A
Cold weather operations	Soil sample stations	Likely	N/A
Heavy manual lifting/moving	Soil cores, moving sample coolers	Likely	N/A
Oxidizers – storage and use	N/A	N/A	N/A
Slips/trips/falls	Soil sample stations	Likely	N/A
Inclement weather – rain, wind	Soil sample stations	Likely	N/A
Sharp objects – machete	N/A	N/A	N/A
Sharp objects – broken glass	Sample jars	Likely	N/A

Table 1

Job Hazard Analysis for Soil Sampling – Types of Potential Hazards

Corrosives - storage and use	N/A	N/A	N/A
Flammable liquids – storage and use	N/A	N/A	N/A
Material handling	Coolers with soil samples	Likely	N/A
Vehicular travel	Van shuttle	Likely	N/A
Working over water	N/A	N/A	N/A
Operational Phase: SJRWP RI/FS	Location: Areas 1, 3, and 4 at the SJRWP; background areas near the SJRWP.		
Biological Hazards			
Name of Biological Hazard	**Source**	**Exposure Level/Potential**	**Exposure Limit**
Ragweed	Beach area	Likely	N/A
Insect bites and stings	Soil sampling areas	Likely	N/A
Operational Phase: SJRWP RI/FS	Location: Areas 1, 3, and 4 at the SJRWP; background areas near the SJRWP.		
Control Measures Used			
Engineering Controls: 1. Use a garden sprayer with potable (or distilled/deionized) water on dry soils to control the generation of dust. 2. Weights of coolers are such that two persons should lift the units to prevent back injuries. 3. To avoid insect bites, personnel will wear long sleeved shirts and mosquito nets if necessary. No insect repellants will be used because they could contaminate samples. 4. Field staff must bring allergy medications if allergic to ragweed. 5. The weight of the impact corer is such that careful lifting and position handling must be observed. 6. To mitigate poisoning from a snake bite, a snake bite kit will be available on Site. Follow the safety procedures regarding snakes that are presented in Section 4.3.3.2. 7. To avoid sinking in mud, mudders will be strapped to boots or pieces of plywood will be used.			
Level of PPE: D	PPE: Steel-toed boots, PVC bib-style overalls (and jacket with hood as necessary), safety glasses or goggles, nitrile gloves. A hard hat will be worn if there are any overhead dangers.		
Work Practices:	Change disposable nitrile gloves frequently. Wash hands and face with soap and water after each sampling event. Take shower at end of workday.		

Notes:
[1] PEL-TWA values from NIOSH Pocket Guide to Chemical Hazards (1997).
[2] TLV-TWA values from American Conference of Governmental Industrial Hygienists (ACGIH 1996).
Inh = Inhalation, Abs = Absorption, Con = Contact, Ing = Ingestion
mg/m^3 = milligrams per cubic meter
NA = Not applicable.
NE = Not established.

If the JHA is modified, then related provisions in other sections of this Soil Sampling HASP will also be modified as needed.

The overall hazard level associated with the activities described in Section 2 is low. Hazards that may be encountered during these sampling programs include physical safety hazards associated with the field operations, exposure to chemicals used to decontaminate sampling gear and preserve samples, and potential exposure to hazardous materials present within the soils. Potential hazards while working at the Site include, but are not limited to, the following:

- Exposure to toxic and/or hazardous chemicals
- Physical hazards from use of sampling equipment
- Physical hazards from working conditions (e.g., hypothermia, slips/trips/falls, or drowning).

As described below, protective equipment and safe working procedures will help prevent accidents caused by these hazards. All workers are required to use the buddy system, and no one will be allowed to work alone.

4.1 Definitions

Chemical hazards are defined by the following terms:

Time-weighted Average (TWA): The recommended exposure limits for a hazardous chemical in the workplace, typically during an 8-hour work day over a 40-hour work week. TWAs are recommended by the National Institute for Occupational Safety and Health (NIOSH) under the authority of OSHA.

Permissible Exposure Limit (PEL): The legal maximum air concentration of a hazardous chemical to which workers may be exposed on an 8-hour basis as established by OSHA. The PEL is a time-weighted average value (PEL-TWA), and for all chemicals discussed below, the corresponding PEL-TWA is the same for OSHA.

Threshold Limit Value (TLV): The recommended maximum air concentration of a hazardous chemical to which workers may be exposed on an 8-hour basis. TLVs are time-weighted average values (TLV-TWA) and are recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).

Short-term Exposure Limit (STEL): A 15-minute TWA exposure that should not be exceeded at any time during a workday.

Ceiling Limit: Employee's exposure, which should not be exceeded during any part of the workday.

Buddy system: "Buddy system" means that an employee is designated to be observed by at least one other employee in the work group. The purpose of the buddy system is to provide rapid assistance to employees in the event of an emergency.

4.2 Chemical Hazards

Table 1 presents a summary of health-based chemical exposure information for the primary COPCs for the 2011 soil sampling. Additional information (including MSDSs and occupational health guidelines) is provided in Appendix B of the overall HASP (Anchor QEA 2009).

4.2.1 Potential Hazards of COPCs in Soils

A summary of the COPCs for health and safety and their concentrations in the Site sediments is provided in Table 1. No soil data are available at the site, so the sediment data were evaluated in this Soil Sampling HASP addendum. The concentrations of COPCs in soils at the site are expected to be similar to those detected in sediments at the Site. No soil data are available for the specific background locations that will be sampled; however, the concentrations are expected to be lower than those at the Site.

The COPC list includes chemicals that were detected in surface sediment samples: dioxins/furans, aluminum, copper, and mercury. During the soil sampling, the surface soils may be dry and pose a risk for inhalation. As a result, engineering controls such as wetting

the soil with potable (or distilled/deionized) water may be necessary to control the generation of dust. Personnel will also be working in an open-air environment. Nonetheless, these compounds are potentially hazardous and exposure by all routes should be minimized. There is no evidence of significant concentrations of volatile chemicals in soil, sediment, or surface water (Anchor QEA 2009). Therefore, respiratory protection is not expected to be needed, and either Level D (off-site sampling handling) or Modified Level D personal protective equipment (PPE) should be appropriate for the entire investigation. MSDSs for these compounds are provided in Appendix B of the overall HASP (Anchor QEA 2009).

4.3 Physical Hazards

The section below provides safety guidelines for the use of vehicles. The different physical hazards that may be associated with these operations are also discussed below.

4.3.1 Motor Vehicle Operation

Motor vehicles will be used to transport field personnel, equipment, and supplies to the nearshore, intertidal sampling locations that will be accessed during low tide. Motor vehicles will also be used to transport field personnel, equipment, and supplies to the sample processing/shipping locations. Only sampling team personnel with valid driver's licenses and liability insurance (per local state laws) will operate motor vehicles required for work activities. All field staff will use best professional judgment at all times to ensure safe operation of motor vehicles, including:

- Operators are to practice defensive driving and drive in a courteous manner
- Operators are to be aware of pedestrians and give them the right-of-way
- All vehicles are to be operated in a safe manner and in compliance with statutory traffic regulations and ordinances
- Operators are to verify that safety seat belts are in proper operating order
- Seat belts are to be worn by the driver and all passengers whenever the vehicle is in motion
- No persons are allowed to ride in the back of any vehicles, unless equipped with seat belts
- Vehicles are to be driven in conformance with local speed limits

- Operators are to avoid excessively long driving periods
- Personnel who are impaired by fatigue, illness, alcohol, illegal or prescription drugs, or who are otherwise physically unfit, are not allowed to drive
- Personnel are to avoid using cellular phones or engaging in other distractions while driving
- Motor vehicle accidents are to be reported to the responsible law enforcement agency, Integral's human resources manager, and Integral's CHSM.

4.3.2 Physical Exposure

Exposure to the elements and fatigue are two major causes of accidents while working outside. The individual task activities may include long work days and unpredictable weather.

To prevent fatigue and overexposure in adverse weather conditions, field personnel will take regular work breaks. Extra clothing will be brought to accommodate changes in weather. Cold stress can be manifested as hypothermia (discussed further in Section 12.2.2 of the overall HASP; Anchor QEA 2009). Heat-related illnesses can occur at any time when protective clothing is worn. When air temperatures average 70 to 75°F, the risk of heat-related illnesses increases. Heat stress can be manifested as both heat stroke and heat exhaustion (discussed further in Section 12.2.1 of the overall HASP; Anchor QEA 2009).

Personnel should monitor their own conditions and capabilities and are responsible for taking appropriate measures to relieve fatigue, exposure, or heat stress. Because fatigue and extreme heat/cold stress may impair an individual's judgment, the FL/SSO is also responsible for monitoring workers' apparent condition in relation to physical exposure. The FL/SSO may direct any crew member to cease working if conditions indicate the potential for overexposure or if overexposure.

4.3.3 Biological Hazards

The following sections describe safety guidelines for alligators and snakes that may be present in the vicinity of the site. Additional information on biological hazards is presented in Section 12.2 of the overall HASP (Anchor QEA 2009).

4.3.3.1 Alligators

Although alligators have not been observed at the site, they are common in the Houston area. Male alligators are large, solitary animals that will defend their territory (NIEHS and OSHA 2010).

Observe the following procedures regarding alligators (NIEHS and OSHA 2010):

- Always be aware of your surroundings. Stay at least 15 feet away from alligators.
- Avoid surprising reptiles.
- Fight back if you are attacked.

4.3.3.2 Snakes

Although snakes have not been observed at the site, the following snakes are common in the Houston area:

- Rattlesnake
- Copperhead snake
- Cottonmouth snake
- Coral snake.

Observe the following procedures regarding poisonous snakes:

- Avoid walking in areas where snakes may nest or hide. When walking, always look for signs of snakes.
- Use extreme caution when moving or lifting objects that could be used by snakes as cover.
- Never reach under or behind objects or into other areas where snakes may hide if you do not have a clear view of the area where your hand and arm will be.
- Wear sturdy leather or firm rubber boots.
- Poisonous snakebites are medical emergencies. If bitten by any type of snake, immediately seek medical attention.

4.3.4 Other Physical Hazards

Incorporating the following basic safety procedures can prevent many of the most common causes of injury or accident during field sampling:

- Always be aware of your surroundings. Transients have been observed to live or spend time in the vicinity of some of the soil sampling stations, both on the Site and in background areas. If you feel threatened, or if the situation feels unpredictable, leave the area immediately. Always use the buddy system when working on the Site and in background areas.
- Implement good housekeeping practices, including immediate cleanup of spills and safe storage of all materials. All equipment or materials not in current use will be removed from the immediate work area.
- Use proper lifting and moving techniques to prevent back or muscle strain or injury. Any heavy equipment, boxes, coolers, or other items should be tested before lifting. If a piece of equipment is too heavy, the equipment should be broken into smaller components or assistance requested. Lifting should be done with the legs, not the back.
- Use extra caution when handling sharp tools or sampling devices and when possible, wear protective gloves.
- Use hearing protection when working with or near a power generator, and when using a circular saw to cut soil cores.
- Use the following safety procedures when employing extension cords:
  - Always inspect cords before using them. Use only cords in good condition to avoid electrical shocks.
  - Extension cords used in wet and/or outdoor locations have to be protected by ground fault circuit interrupters.
  - Extension cords should be a minimum of 16 American Wire Gauge size (AWG) and be rated for the equipment in use. Example: To connect an impact corer to a 2000-watt power generator, a 12 AWG (25 amps) extension cord is needed to carry the necessary current to start up the unit.

- Avoid running extension cords across walkways. Instead, run them overhead if possible and place flagging tape on the extension cord to warn of possible overhead hazard.
- An extension cord that is hot to the touch is overloaded and should be replaced.

4.4 Employee Notification of Hazards and Overall Site Information Program

The information in the JHA and the MSDSs will be made available to all employees who could be affected by it prior to the time they begin their work activities. Modifications to JHAs and the accompanying data sheets will be communicated during routine briefings.

Consistent with paragraph 1910.120 (i) of Hazardous Waste Operations and Emergency Response (HAZWOPR) (OSHA 1994), the FL/SSO will also inform other contractors and subcontractors working on this study about the nature and level of hazardous substances at the Site, the likely degree of exposure to workers who participate in Site operations, and any modifications to this Soil Sampling HASP.

Daily safety briefings will take place before work begins. The daily briefing form provided in Exhibit 1 will be used to record the daily meetings.

5 SITE CONTROL ZONES

The definitions of the Site control zones are discussed in Section 7.3 of the overall HASP (Anchor QEA 2009). The use of Site control zones is designed to reduce the spread of hazardous substances from contaminated areas to clean areas, to identify and isolate contaminated areas of the Site, to facilitate emergency evacuation and medical care, to prevent unauthorized entry to the Site, and to deter vandalism and theft.

5.1 Exclusion Zone

The exclusion zone consists of a 10-foot radius around each soil sample station. The exclusion zone will be marked with caution tape or traffic safety cones.

5.2 Contamination Reduction Zone

Not applicable. All activities will take place within the exclusion zone.

5.3 Support Zone

All areas outside the exclusion zone.

6 PROJECT AIR MONITORING REQUIREMENTS

Section 11 of the overall HASP (Anchor QEA 2009) provides general requirements for air monitoring during the 2011 soil study, including information on air monitoring equipment. Previous investigations of the Site indicate that the main chemicals of interest for worker health and safety during the sampling event(s) are dioxins and furans. There is no evidence of significant concentrations of volatile chemicals in soil, sediment, or surface water. Therefore, respiratory protection is not expected to be needed and either Level D (off-site sample handling) or Level Modified D PPE (sampling activities) will be used.

7 DECONTAMINATION OF SAMPLING EQUIPMENT

Decontamination of sampling equipment will follow procedures in Section 2.1.5 of the field sampling plan. No chemical solvents will be required for decontamination of sampling equipment.

All vehicles and equipment that have entered potentially contaminated areas will be visually inspected and, if necessary, decontaminated prior to leaving the area by rinsing tires and wheel wells with Liquinox® or Alconox® detergent and water. An effort will be made to keep vehicles away from contaminated soil and sediment by parking on the service road and carrying field sampling equipment to the Site on foot or by using carts or sleds. Large tools will be cleaned in the same manner. Small reusable sampling equipment, including bowls, spoons, and knives, will be rinsed, washed in phosphate-free detergent, and rinsed again. All personnel walking over the impoundment area will have their boots decontaminated as well. Rinsate from all decontamination activities will be collected for proper disposal. Decontamination of equipment and tools will take place within the support/contamination reduction zone.

The following supplies will be available to perform decontamination activities:

- Wash and rinse buckets
- Tap water and phosphate-free detergent (i.e., Alconox® or Liquinox®)
- Scrub brushes
- Distilled/deionized water
- Pressure washer/steam cleaner, if appropriate
- Paper towels and plastic garbage bags
- 55-gallon drums with labels and lids or 5-gallon plastic buckets with labels and lids to segregate rinsed waste water and solid waste derived from soil sampling and processing activities.

8 REFERENCES

ACGIH, 1996. Threshold Limit Values (TLV) for Chemical Substances and Physical Agents Biological Exposure Indices (BEIs). American Conference of Governmental Industrial Hygienists, Cincinnati, OH.

Anchor QEA, 2009. Health and Safety Plan San Jacinto River Waste Pits Superfund Site. Prepared for McGinnes Industrial Maintenance Corporation, International Paper Company, and U.S. Environmental Protection Agency, Region 6. Anchor QEA, LLC, Ocean Springs, MS.

NIEHS and OSHA, 2010. Safety and Health Awareness for Oil Spill Cleanup Workers, Worker Education & Training Program. U.S. Department of Health and Human Services, National Institute of Environmental Health Sciences and Department of Labor, Occupational Safety and Health Administration. OSHA 3388-062010. June 2010.

NIOSH, 1997. National Institute for Occupational Safety and Health (NIOSH) Pocket Guide to Chemical Hazards. Keller & Associates, Neenah, WI.

OSHA, 1994. 29 CFR Parts 1910 and 1926. Hazardous Waste Operations and Emergency Response. Department of Labor, Occupational Safety and Health Administration. Accessed web site on January 8, 2010. http://www.osha.gov/pls/oshaweb/owasrch.search_form?p_doc_type=STANDARDS&p_toc_level=1&p_keyvalue=1910

OSHA, 1997. EPA/Labor Superfund Health & Safety Task Force: OSHA Audits of Superfund Sites from 1993 to 1996. Department of Labor, Occupational Safety and Health Administration. August 25, 1997. Accessed web site on January 8, 2010. http://www.osha.gov/SLTC/hazardouswaste/sftaskrpt.html

ATTACHMENT A2

STANDARD OPERATING PROCEDURES

LIST OF STANDARD OPERATING PROCEDURES

SOP AP-01 Sample Packaging and Shipping

SOP AP-02 Field Documentation

SOP AP-03 Sample Custody

SOP AP-04 Sample Labeling

SOP AP-06 Navigation and Station Positioning

SOP SL-01 Decontamination of Soil Sampling Equipment

SOP SL-02 Preparation of Field Quality Control Samples for Soils

SOP SL-04 Soil Classification

SOP SL-05 Surface Soil Sampling

SOP SL-06 Logging of Soil Boreholes

STANDARD OPERATING PROCEDURE (SOP) AP-01

SAMPLE PACKAGING AND SHIPPING

SCOPE AND APPLICATION

This SOP describes specific requirements for sample packaging and shipping to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein. This SOP also presents the method to be used when packing samples that will either be hand delivered or shipped by commercial carrier to the laboratory.

EQUIPMENT AND SUPPLIES REQUIRED

Make sure that you have the equipment and supplies necessary to properly pack and ship environmental samples, including the following:

- Project-specific sampling and analysis plan (SAP)
- Project-specific field logbook
- Sealable airtight bags in assorted sizes (e.g., Ziploc®)
- Wet ice in doubled, sealed bags; frozen Blue Ice®; or dry ice
- Cooler(s)
- Bubble wrap
- Fiber-reinforced packing tape, clear plastic packing tape, and duct tape
- Scissors or knife
- Chain-of-custody (COC) forms
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick)
- Paper towels
- "Fragile," "This End Up," or "Handle With Care" labels
- Mailing labels
- Air bills for overnight shipment

PROCEDURE

Customize the logistics for sample packaging and shipping to each study. If necessary, transfer samples from the field to a local storage facility where they can be frozen or refrigerated. Depending on the logistics of the operation, field personnel may transport samples to the laboratory or use a commercial courier or shipping service. In the latter case, Integral field personnel must be aware of any potentially limiting factors to timely shipping, such as availability of overnight service and weekend deliveries to specific areas, and shipping regulations regarding "restricted articles" (e.g., dry ice, formalin) prior to shipping the samples.

SAMPLE PREPARATION

Take the following steps to ensure the proper transfer of samples from the field to the laboratories:

At the sample collection site:

1. Document all samples using the proper logbooks or field forms (see SOP AP-02), required sample container identification (i.e., sample labels with tag numbers), and COC form (example provided in SOP AP-03). Fill out the COC form as described in SOP AP-03, and use the sample labeling techniques provided in SOP AP-04.
2. Make all applicable laboratory quality control sample designations on the COC forms. Clearly identify samples that will be archived for future possible analysis. Label these samples as follows: "Do Not Analyze: Hold and archive for possible future analysis." Some laboratories interpret "archive" to mean that they should continue holding the residual sample after analysis.
3. Notify the laboratory contact and the Integral project quality assurance/quality control (QA/QC) coordinator that samples will be shipped and the estimated arrival time. Send copies of all COC forms to Integral's project QA/QC coordinator or project manager, as appropriate.
4. Keep the samples in the possession of the sampling personnel at all times. Lock and secure any temporary onsite sample storage areas to maintain sample integrity and COC requirements.
5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
6. Complete the COC form as described in SOP AP-03, and retain the back (pink) copy for project records prior to sealing the cooler. Check sample containers against the COC form to ensure all the samples that were collected are in the cooler.

7. Store each sample container in a sealed plastic bag that allows the sample label (example provided in SOP AP-03) to be read. Before sealing the bags, ensure that volatile organic analyte (VOA) vials are encased in a foam sleeve or in bubble wrap.
8. If the samples require storage at a specific temperature, place enough ice in the sample cooler to maintain the temperature (e.g., 4°C) throughout the sampling day.

At the sample processing area (immediately after sample collection) take the following steps:

1. If the samples require a specific storage temperature, then cool the samples and maintain the temperature prior to shipping. For example, place enough ice in each sample cooler to maintain the temperature at 4°C until processing begins at the testing laboratory.
2. Be aware of holding time requirements for project-specific analytes and arrange the sample shipping schedule accordingly.
3. Place samples in secure storage (i.e., locked room or vehicle) or keep them in the possession of Integral sampling personnel before shipment. Lock and secure any sample storage areas to maintain sample integrity and COC requirements.
4. Store samples in the dark (e.g., keep coolers shut).

At the sample processing area (just prior to shipping), do the following:

1. Check sample containers against the COC form to account for all samples intended for shipment.
2. Choose cooler(s) of appropriate size and make sure they are clean of gross contamination inside and out. If the cooler has a drain, close the drain and secure it with duct tape.
3. Line the cooler with bubble wrap and place a large plastic bag (preferably with a thickness of 3 mil), open, inside the cooler.
4. Individually wrap each glass container (which was sealed in a plastic bag at the collection site) in bubble wrap and secure with tape or a rubber band. Place the wrapped samples in the large plastic bag in the cooler, leaving room for ice to keep the samples cold (i.e., 4°C).
5. If temperature blanks have been provided by the testing laboratory, place one temperature blank in each sample cooler.
6. If the samples require a specific storage temperature, add enough wet ice or Blue Ice® to maintain that temperature during overnight shipping (i.e., 4°C). Always overestimate the amount of ice that will be required. Keep ice in a sealed plastic bag, which is placed in a second sealed plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it may insulate the samples from the ice. After adding all samples and ice to the cooler, use bubble wrap (or other

available clean packing material) to fill any empty space and prevent the samples from shifting during transport.

7. If possible, consolidate all VOA samples in a single cooler and ship them with (a) trip blank(s) if the project-specific QA project plan calls for them.
8. Sign, date, and include any tracking numbers provided by the shipper on the COC form. Remove the back (pink) copy of the original COC form and retain this copy for the project records.
9. Seal the rest of the signed COC form in a bag and tape the bag to the inside of the cooler lid. Each cooler should contain an individual COC form for the samples contained inside it. If time is short and it becomes necessary to combine all the samples onto a single set of COC forms and ship multiple coolers together, then indicate on the outside of the appropriate cooler, "Chain-of-Custody Inside."
10. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it with fiber-reinforced packing tape. Tape the cooler around the opening, joining the lid to the bottom, and around the circumference of the cooler at both hinges.
11. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid (provided with example field forms). Place one seal on the front right portion of the cooler and one on the back left. Be sure the seals are properly affixed to the cooler to prevent removal during shipment. Additional tape across the seal may be necessary if the outside of the cooler is wet.

SAMPLE SHIPPING

Hand Delivery to the Testing Laboratory

1. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. When hand-delivering environmental samples, make sure the testing laboratory receives them on the same day that they were packed in the coolers.
3. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after it has been faxed. Never leave the original COC form in the custody of non-Integral staff.

Shipped by Commercial Carrier to the Laboratory

1. Apply a mailing label to the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care." If the shipment contains multiple coolers, indicate on the mailing label the number of coolers that the testing laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the cooler and to protect it from the weather. This is a secondary label in case the air bill is lost during shipment.
2. Fill out the air bill and fasten it to the handle tags provided by the shipper (or the top of the cooler if handle tags are not available).
3. If samples must be frozen (–20°C) during shipping, make sure that dry ice has been placed in the sample cooler. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require.
4. Make sure that benthic infauna samples have been preserved with formalin in the field prior to shipping. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require for these samples.
5. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be shipped and the estimated arrival date and time. If environmental samples must be shipped at 4°C or –20°C, choose overnight shipping for delivery next morning. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after faxing. Never leave the original COC form in the custody of non-Integral staff.

STANDARD OPERATING PROCEDURE (SOP) AP-02

FIELD DOCUMENTATION

SCOPE AND APPLICATION

This SOP describes the Integral procedure for accurate record-keeping in the field for the purposes of ensuring that samples can be traced from collection to final disposition.

Document all information relevant to field operations properly to ensure that activities are accounted for in written records to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. Several types of field documents are used for this purpose and should be consistently used by field personnel. Field documentation should include only a factual description of site-related activities and observations. Field personnel should not include superfluous comments or speculation regarding the field activities or observations.

FIELD LOGBOOKS

During field sampling events, field logbooks must be used to record all daily activities. The purpose of the field logbook is to document events and record data measured in the field to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. The project manager (or designee) should issue a field logbook to the appropriate site personnel for the direction of onsite activities (e.g., reconnaissance survey team leader, sampling team leader). It is this designee's responsibility to maintain the site logbook while it is in his or her possession and return it to the project manager or turn it over to another field team.

Make entries in the field logbook as follows:

1. Document all daily field activities in indelible ink in the logbook and make no erasures. Make corrections with a single line-out deletion, followed by the author's initials and the date. The author must initial and date each page of the field logbook. The author must sign and date the last page at the end of each day, and draw a line through any blank space remaining on the page below the last entry.

2. Write the project name, dates of the field work, site name and location (city and state), and Integral job number on the cover of the field logbook. If more than one logbook is used during a single sampling event, then annotate the upper right-hand corner of the logbook (e.g., Volume 1 of 2, 2 of 2) to indicate the number of logbooks used during the field event. Secure all field logbooks when not in use in the field. The following is a list of the types of information that is appropriate for entry in the field notebook:
   - Project start date and end date
   - Date and time of entry (24-hour clock)
   - Time and duration of daily sampling activities
   - Weather conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change (e.g., wind speed and direction, rain, thunder, wave action, current, tide, vessel traffic, air and water temperature, thickness of ice if present)
   - Name and affiliation of person making entries and other field personnel and their duties, including what times they are present
   - The location and description of the work area, including sketches, map references, and photograph log, if appropriate
   - Level of personal protection being used
   - Onsite visitors (names and affiliations), if any, including what times they are present
   - The name, agency, and telephone number of any field contacts
   - Notation of the coordinate system used to determine the station location
   - The sample identifier and analysis code for each sample to be submitted for laboratory analysis, if not included on separate field data sheets
   - All field measurements made (or reference to specific field data sheets used for this purpose), including the time of collection and the date of calibration, if appropriate
   - The sampling location name, date, gear, water depth (if applicable), and sampling location coordinates, if not included on separate field data sheets
   - For aquatic sampling, the type of vessel used (e.g., size, power, type of engine)
   - Specific information on each type of sampling activity
   - The sample type (e.g., groundwater, soil, surface sediment), sample number, sample tag number, and any preservatives used, if not included on separate field data sheets
   - Sample storage methods

- Cross-references of numbers for duplicate samples
- A description of the sample (source and appearance, such as soil or sediment type, color, texture, consistency, presence of biota or debris, presence of oily sheen, changes in sample characteristics with depth, presence/location/thickness of the redox potential discontinuity [RPD] layer, and odor) and penetration depth, if not included on separate field data sheets
- Estimate of length and appearance of recovered cores, if not included on separate field data sheets
- Photographs (uniquely identified) taken at the sampling location, if any
- Details of the work performed
- Variations, if any, from the project-specific sampling and analysis plan (SAP) or standard operating protocols and reasons for deviation
- Details pertaining to unusual events that might have occurred during sample collection (e.g., possible sources of sample contamination, equipment failure, unusual appearance of sample integrity, control of vertical descent of the sampling equipment)
- References to other logbooks or field forms used to record information (e.g., field data sheets, health and safety log)
- Any field results not appearing on the field data sheets (if used), including station identification and location, date, and time of measurement
- Sample shipment information (e.g., shipping manifests, chain-of-custody (COC) form numbers, carrier, air bill numbers, time addresses)
- A record of quantity of investigation-derived wastes (if any) and storage and handling procedures.

3. During the field day, as listed above, record in the logbook a summary of all site activities. Provide a date and time for each entry. The information need not duplicate anything recorded in other field logbooks or field forms (e.g., site health and safety officer's logbook, calibration logbook, field data sheets), but should summarize the contents of the other logbooks and refer to the pages in these logbooks for detailed information.
4. If measurements are made at any location, record the measurements and equipment used, or refer to the logbook and page number(s) or field forms on which they are recorded. All maintenance and calibration records for equipment should be traceable through field records to the person using the instrument and to the specific piece of instrumentation itself.

5. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided below.

FIELD DATA FORMS

Occasionally, additional field data forms are generated during a field sampling event (e.g., groundwater monitoring form, sediment core profile form, water quality measurement form) to record the relevant sample information collected. For instructions regarding the proper identification of field data forms, sampling personnel should consult the project-specific SAP.

Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field data forms to be copied. A discussion of copy distribution is provided below.

PHOTOGRAPHS

In certain cases, photographs (print or digital) of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Ensure that photographs include a measured scale in the image, when practical. If you take photographs of sample characteristics and routine sampling activities, avoid using telephoto or wide-angle shots, because they cannot be used in enforcement proceedings. Record the following items in the field logbook for each photograph taken:

1. The photographer's name or initials, the date, the time of the photograph, and the general direction faced (orientation)
2. A brief description of the subject and the field work shown in the picture
3. For print photographs, the sequential number of the photograph and the roll number on which it is contained
4. For digital photographs, the sequential number of the photograph, the file name, the file location, and back-up disk number (if applicable).

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all photographic materials to be developed (prints) or copied (disks). Place the prints or disks and associated negatives in the project files (at the Integral project manager's location). Make photocopies of photo logs and any supporting documentation from the field logbooks, and place them in the project files with the prints or disks.

EQUIPMENT CALIBRATION RECORDS

Record in the field logbook all equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration. Calibrate all equipment used during the investigation daily, at a minimum, in accordance with the manufacturers' recommendations.

DISTRIBUTION OF COPIES

At Integral offices, make two copies of all field logbooks and additional field data forms. Stamp the first copy with a "COPY" stamp, and place it in the project file to be available for general staff use. Stamp the second copy with a "FILE" stamp, and place it in the data management file with the laboratory data packages, to be used by the data management and quality assurance staff only. Place the original field logbooks and forms in a locked file cabinet.

SET-UP OF LOCKING FILE CABINET

Place each project in its own file folder in a locking file cabinet. On the folder label, include the project name and contract number. Each project folder will include up to six kinds of files:

- Field logbook(s)
- Additional field data forms
- Photographs
- COC forms
- Acknowledgment of Sample Receipt forms
- Archive Record form (to be completed only if samples are archived at an Integral field storage facility or Integral laboratory).

STANDARD OPERATING PROCEDURE (SOP) AP-03

SAMPLE CUSTODY

SCOPE AND APPLICATION

This SOP describes Integral procedures for custody management of environmental samples.

A stringent, established program of sample chain-of-custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used with SOP AP-01, which covers sample packaging and shipping; SOP AP-02, which covers the use of field logbooks and other types of field documentation; and SOP AP-04, which covers sample labeling. Chain-of-custody (COC) forms ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession
2. The sample is in the person's view after being in his or her possession
3. The sample is in the person's possession and is being transferred to a designated secure area
4. The sample has been locked up to prevent tampering after it was in the person's possession.

At no time is it acceptable for samples to be outside of Integral personnel's custody unless the samples have been transferred to a secure area (i.e., locked up). If the samples cannot be placed in a secure area, then an Integral field team member must physically remain with the samples (e.g., at lunch time one team member must remain with the samples).

CHAIN-OF-CUSTODY FORMS

The COC form is critical because it documents sample possession from the time of collection through final disposition. The form also provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

Complete the COC form after each field collection activity and before shipping the samples to the laboratory. Sampling personnel are responsible for the care and custody of the samples until they are shipped. The individuals relinquishing and receiving the samples must sign the

COC form(s), indicating the time and date of the transfer, when transferring possession of the samples.

A COC form consists of three-part carbonless paper with white, yellow, and pink copies. The sampling team leader keeps the pink copy. The white and yellow sheets are placed in a sealed plastic bag and secured inside the top of each transfer container (e.g., cooler). Field staff retain the pink sheet for filing at the Integral project manager's location. Each COC form has a unique four-digit number. This number and the samples on the form must be recorded in the field logbook. Integral also uses computer-generated COC forms. If computer-generated forms are used, then the forms must be printed in triplicate and all three sheets signed so that two sheets can accompany the shipment to the laboratory and one sheet can be retained on file. Alternatively, if sufficient time is available, the computer-generated forms will be printed on three-part carbonless paper.

Record on the COC form the project-assigned sample number and the unique tag number at the bottom of each sample label. The COC form also identifies the sample collection date and time, type of sample, project name, and sampling personnel. In addition, the COC form provides information on the preservative or other sample pretreatment applied in the field and the analyses to be conducted by referencing a list of specific analyses or the statement of work for the laboratory. The COC form is sent to the laboratory along with the sample(s).

PROCEDURES

Use the following guidelines to ensure the integrity of the samples:

1. Sign and date each COC form. Have the person who relinquishes custody of the samples also sign this form.
2. At the end of each sampling day and prior to shipping or storage, make COC entries for all samples. Check the information on the labels and tags against field logbook entries.
3. Do not sign the COC form until the team leader has checked the information for inaccuracies. Make corrections by drawing a single line through any incorrect entry, and then initial and date it. Make revised entries in the space below the entries. After making corrections, mark out any blank lines remaining on the COC form, using single lines that are initialed and dated. This procedure will prevent any unauthorized additions.

   At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date of the transfer. The time the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as FedEx or United Parcel Service (UPS), record the name of the carrier on the COC form. Also enter on the COC form any tracking numbers supplied by the carrier. The time of transfer should be as close to the actual drop-off time as possible. After signing the COC forms and removing the pink copy, seal them inside the transfer container.
5. If errors are found after the shipment has left the custody of sampling personnel, make a corrected version of the forms and send it to all relevant parties. Fix minor errors by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.
6. Provide a COC form and an Archive Record form for any samples that are archived internally at Integral.

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all COC forms to be copied. A discussion of copy distribution is provided in SOP AP-02.

CUSTODY SEAL

As security against unauthorized handling of the samples during shipping, affix two custody seals to each sample cooler. Place the custody seals across the opening of the cooler (front right and back left) prior to shipping. Be sure the seals are properly affixed to the cooler so they cannot be removed during shipping. Additional tape across the seal may be prudent.

SHIPPING AIR BILLS

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), the shipper provides an air bill or receipt. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting the sender's copy of all shipping air bills to be copied at an Integral office. A discussion of copy distribution is provided in SOP AP-02. Note the air bill number (or tracking number) on the applicable COC forms or, alternatively, note the applicable COC form number on the air bill to enable the tracking of samples if a cooler becomes lost.

ACKNOWLEDGMENT OF SAMPLE RECEIPT FORMS

In most cases, when samples are sent to a testing laboratory, an Acknowledgment of Sample Receipt form is faxed to the project QA/QC coordinator the day the samples are received by the laboratory. The person receiving this form is responsible for reviewing it, making sure that the laboratory has received all the samples that were sent, and verifying that the correct analyses were requested. If an error is found, call the laboratory immediately, and document

any decisions made during the telephone conversation, in writing, on the Acknowledgment of Sample Receipt form. In addition, correct the COC form and fax the corrected version to the laboratory.

Submit the Acknowledgment of Sample Receipt form (and any modified COC forms) to be copied. A discussion of copy distribution is provided in SOP AP-02.

ARCHIVE RECORD FORMS

On the rare occasion that samples are archived at an Integral office, it is the responsibility of the project manager to complete an Archive Record form. This form is to be accompanied by a copy of the COC form for the samples, and will be placed in a locked file cabinet. The original COC form remains with the samples in a sealed Ziploc® bag.

STANDARD OPERATING PROCEDURE (SOP) AP-04

SAMPLE LABELING

SCOPE AND APPLICATION

This SOP describes the general Integral procedures for labeling samples, and the three kinds of labels that can be used on a project (i.e., sample labels, sample tags, and internal sample labels). Consult the project-specific sampling and analysis plan (SAP) to determine the exact sample identifiers and sample labels that are required for a given project. If they are not specified in the SAP, then follow the designations below.

SAMPLE IDENTIFIERS

Before field sampling begins, establish sample identifiers to be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., replicates) to ensure proper data analysis and interpretation, 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples, and 3) to track individual sample containers to ensure that the laboratory receives all material associated with a single sample. To accomplish these purposes, each container may have three different codes associated with it: the sample identifier, the sample number, and the sample tag number. These codes and their use are described as follows:

- **Sample Identification Code**—The sample identification code (Sample ID) is a unique designation that identifies where and how the sample was collected. The sample identifier is recorded in the field logbook *only* and is not provided on the sample label or chain-of-custody (COC) form. The sample identifier is a multiple-part code. The first component begins with the letter abbreviation; for example, "SWNS" or "SWNB" to designate the surface water sample was collected from the near-surface or near-bottom of the water column. The second part could identify the sampling event; for example, "1" to designate Round 1 sampling. The third part could contain an abbreviation for whether the station is a single point (SP), a transect (TR), a composite (CO), or a vertically integrated station (VI). The station number would be the final component of the sample identifier. Use leading zeros for stations with numbers below 100 for ease of data management and correct data sorting.

  If appropriate, add a supplemental component to the sample identifier to code field

duplicate samples and splits. Use a single letter (i.e., a suffix of "A" and "B") to indicate field duplicates or splits in the final component of the sample identifiers. For equipment decontamination blanks, assign sequential numbers starting at 900 instead of station numbers. Use a sample type code that corresponds to the sample type for which the decontamination blank was collected. Additional codes may be adopted, if necessary, to reflect sampling equipment requirements (see project-specific SAP).

Examples of sample IDs are as follows:

- SWNS-1-SP-002: Surface water sample collected from the near-surface at a single point during Round 1 from Station 2.
- SWNB-1-TR-010-A: Duplicate surface water sample from the near-bottom transect during Round 1 from Station 10.

- **Sample Number**—The sample number is an arbitrary number assigned to each distinct sample or split that is shipped to the laboratory for separate analysis. The sample number appears on the sample containers and the COC forms. Each sample will be assigned a unique sample number. All aliquots of a composited field sample will have the same sample number. In cases where samples consist of multiple bottles from the same location, assign each bottle the same sample number and time. However, assign replicates from the same location different sample numbers and times. Sample numbers of related field replicates will not necessarily have any shared content.

  Each field split of a single sample will also have a different sample number and time. The sample number is generally a unique six-digit number that includes a two-digit media code and a four-digit number. The media code may be site-specific, but the Integral default codes are as follows:

  - SS—Surface soil
  - BH—Subsurface soil or rock (typically from borehole)
  - GW—Groundwater
  - SW—Surface water
  - PW—Pore water
  - SD—Sediment
  - BT—Biota or biological tissue

    The exact sample numbering scheme may vary from project to project. Variances in the sample numbering scheme will be described in the project-specific SAP for the field event. Example sample numbers are PW0001, PW0002, PW0003, etc.

- **Tag Number—**Attach a different tag number to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, assign each container the same sample number and a different sample tag. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted).

  The sample tag number is a unique five- or six-digit number assigned to each sample label (or "tag") for multiple bottles per sample. Integral sample labels come with a preprinted sample tag number. The tag number provides a unique tracking number to a specific sample bottle. This allows for greater flexibility in tracking sample bottles and assists in field quality control when filling out documentation and shipping. Sample tags are not used by many other consultants, and there may be resistance from such firms during teaming situations. However, experience has shown that tags can be very valuable, both in the field and while processing data from field efforts.

  Record tag numbers on the COC form. Laboratories use tag numbers only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Assign sample numbers sequentially in the field; sample labels are preprinted with sequential tag numbers.

SAMPLE LABELS

Integral sample labels are designed to uniquely identify each individual sample container that is collected during a sampling event. Field sampling teams are provided with preprinted sample labels, which must be affixed to each sample container used. Fill out the labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- A unique number (commonly referred to as the "Tag Number") that is preprinted on the label consisting of five or six digits; used to identify individual containers.

SAMPLE TAGS

Integral sample tags are designed to be affixed to each container that is used for a sample. Sample tags are required only for environmental samples collected in certain U.S.

Environmental Protection Agency (EPA) regions (e.g., EPA Region 5). Field crews are provided with preprinted sample tags. Attach sample tags to each individual sample container with a rubber band or wire through a reinforced hole in the tag. Mark all sample tag entries with indelible ink. Fill out the tags at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- Type of analysis.

A space for the laboratory sample number (provided by the laboratory at log-in) will also be provided on the sample tag.

INTERNAL SAMPLE LABELS

For benthic infaunal samples, wash away the sediment from the sample and collect the remaining benthic infauna into a sample container. Affix sample label (as discussed above) to the outside of the sample container. In addition, place an internal sample label inside the sample container. This internal sample label is made of waterproof paper; be sure to make all internal sample label entries with pencil. Fill out the internal sample labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservative used (e.g., formalin).

STANDARD OPERATING PROCEDURE (SOP) AP-06

NAVIGATION AND STATION POSITIONING

SCOPE AND APPLICATION

This SOP describes procedures for accurate station positioning required to ensure quality and consistency in collecting samples and in data interpretation and analysis. Station positioning must be both absolutely accurate in that it correctly defines a position by latitude and longitude, and relatively accurate in that the position must be repeatable, allowing field crew to reoccupy a station location in the future (e.g., for long-term monitoring programs).

This SOP describes the most commonly used station positioning method, differential global positioning system (DGPS). Integral uses a Trimble Pathfinder™ Pro XRS DGPS for station positioning for many field efforts. The Pro XRS offers the submeter accuracy often required for documenting sampling station locations and for re-locating previously sampled stations. A comprehensive discussion of the Trimble Pathfinder™ Pro XRS DGPS is provided in Attachments 1, 2, and 3 of this SOP.

SUMMARY OF METHOD

Global positioning system (GPS) navigation is used to position the sampler at the desired location. GPS is a satellite-based system that receives positioning data at 1-second intervals from multiple satellites at known positions in space. Standard GPS is calculated to an accuracy of about 10 m.

One can obtain a higher accuracy of approximately 2 m by applying differential corrections to the standard GPS positioning data using DGPS. These differential corrections are applied by sending GPS differential corrections to the GPS receiver via radio transmission. If the sampling location is near the coastal U.S, the U.S. Coast Guard generates differential corrections that are transmitted via radio link to the GPS receiver. If a Coast Guard station is out of range of the sampling area, then a receiver may be set up at a known (i.e., surveyed) reference point on land, or real-time satellite differential signals can be purchased from a private company (e.g., OmniSTAR).

With the Pro XRS, GPS data can be gathered to submeter accuracy using a choice of differential correction sources (i.e., free beacon differential signals such as Coast Guard beacons or OmniSTAR) without establishing a reference station. Data must be corrected to gain submeter accuracy. Free beacon or base station signals allow differential corrections to be

performed after data collection by using a nearby beacon or base station logging data files. (Note: The station must be within 300 miles of the data collection location.) For satellite-based signals, a built-in virtual base station allows for real-time data correction, eliminating the need for post-processing data in some cases. However, postprocessing data corrections can obtain accuracies in the range of 30–50 cm. These accuracies are for the horizontal (northing and easting) component only. The vertical component (elevation) accuracy ranges from submeter to 3 times larger than the horizontal accuracy.

The GPS receiver displays and transmits differentially corrected positioning data to the computer using an integrated navigation software package (e.g., HYPACK, Terrasync). The computer data are typically displayed and recorded in World Geodetic System of 1984 (WGS-1984) geographic coordinates (latitude/longitude). However, the integrated navigation system can display and record information in other datums (e.g., UTM, NAD83). The integrated navigation system, acting as a data manager, displays the sampler's position relative to a target station location in plan view on a video screen. The resulting pictorial screen presentation, as well as numeric navigation data (e.g., range and bearing to the target sampling location) assists the vessel operator (when sampling on-water) in approaching and maintaining the station position while sampling.

SUPPLIES AND EQUIPMENT

- Cable
- GPS antenna
- Telemetry antenna (for differential corrections)
- GPS receiver
- Differential corrections receiver
- Computer and monitor
- Navigation software (e.g., Terrasync)
- Logbook or log sheets.

PROCEDURES

Obtain latitude and longitude coordinates at the locations where samples are collected. An average positioning objective is to accurately determine and record the positions of all sampling locations to within 2 m. Positioning accuracies on the order of 1–3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS provides the operator with a listing of the time intervals during the

day when accuracies are decreased. Avoiding these times allows for better positioning accuracy.

On-Land Sampling Event

A backpack DGPS unit may be used to direct the sampling team to the proposed sampling location. To expedite field activities, enter the target station coordinates into the navigation system database prior to beginning sampling. Place the DGPS antenna as close as possible to where the sampling will occur. Once the sample(s) have been collected at the appropriate location, record the horizontal coordinates of the station in the field logbook.

On-Water Sampling Event

Mount the GPS antenna vertically at the outboard end of the vessel's boom, with the GPS antenna cable extended along the boom into the cabin. Mount the telemetry antenna for receiving differential corrections on a convenient fixture outside the cabin. Locate the GPS receiver, the differential corrections receiver, and the computer in the cabin. Orient the video screen for the computer to allow the vessel operator to observe on-screen positioning data from the helm.

Alternatively, use a backpack DGPS unit to position the sampling vessel (e.g., barge) over a proposed sampling location. Place the DGPS beacon as close as possible to where the drilling will occur (i.e., moon pool). Using the DGPS unit, direct the sampling vessel operator to the sample station location.

Once the sampling vessel is anchored at the appropriate location, record the horizontal coordinates of the station in the field logbook. To expedite field activities, enter the target station coordinates in the navigation system database prior to beginning sampling.

Positioning System Verification

GPS requires no calibration, as all signal propagation is controlled by the U.S. government (the Department of Defense for satellite signals and the U.S. Coast Guard for differential corrections). Verifying the accuracy of the GPS requires coordinates to be known for one (or more) horizontal control point within the study area. The GPS position reading at any given station can then be compared to the known control point. Verify the GPS accuracy at the beginning and end of each sampling day.

Station Positioning Activities

Use a consistent routine for each day's positioning activities. After confirming successful reception of differential signals, turn on the computer on, and the boot the software. Verify the accuracy of the system at a horizontal control point, as described in the previous section.

The sampling team proceeds to a target station location selected by the team leader. That station location is then selected from a number of preselected station locations that have been entered into the integrated navigation system database. Once the station has been selected, the positioning data are displayed on the computer screen or hand-held unit to assist in proceeding to the station and in maintaining the station position during sampling. A confirmed position is recorded electronically each time a sample collection is attempted. (This means that during sediment grab sampling and coring, the locations of both accepted and rejected grabs or cores are recorded.) Upon recovery of the sampling device, read the station position northing (y) and easting (x) coordinates from the archived computer file and record them in the field logbook or on log sheets as a backup to the computer record. Also record time and water depth, if applicable. Ancillary information recorded in the field logbook may include personnel operating the GPS, tidal phase, type of sampling activity, and time when coordinates were collected.

REFERENCES

Trimble Navigation Limited. 2001. TSC1 Asset Surveyor operation manual. Version 5.20. http://trl.trimble.com/dscgi/ds.py/Get/File-8145/Oper.pdf.

Trimble Navigation Limited. 2007. GPS tutorial. Accessed on January 12, 2007. http://www.trimble.com/gps/index.shtml.

ATTACHMENT 1
PRO XRS DESCRIPTION

The Pro XRS combines a high-performance GPS receiver and antenna, beacon differential receiver, and satellite differential receiver in one compact unit. It also includes Trimble's advanced Everest™ technology, which allows users to collect accurate position data near walls, water, vehicles, or other surfaces that reflect satellite signals. Reflected signals, also called multipath signals, make it difficult for GPS receivers to accurately determine position. Everest™ uses a patented technique to remove multipath signals before measurements are used to calculate position.

Equipment Required

The GPS Pathfinder™ Pro XRS consists of the following:

- GPS receiver in backpack casing (with system batteries and cables)
- Hand-held data logger (TSC1) and cable, *or* laptop computer with Terrasync software installed and cable. (Note: Terrasync procedures are described under separate cover.)
- Pro XRS antenna, range poles, and cable
- Compass and tape measure
- Spare 12-volt camcorder and 9-volt batteries (minimum of two each) (use only Kodak, Duracell, or Energizer 9-volt batteries)
- Battery charger and power cord.

Pro XRS Setup

Follow these procedures for the proper setup of the Pro XRS:

1. Ensure that connections between batteries, receiver, and data logger are correct and secure. The coaxial antenna cable connects from the GPS receiver port "ANT" to the base of the antenna. The TSC1 cable (a "pig-tail"-type cable) connects from the bottom or top of the TSC1 to the receiver port "B," where a 9-pin serial port dongle is attached. The dual Y-clip cables should be connected from the receiver to the batteries. Alternatively, if AC power is available (e.g., aboard a vessel), then the power cable for the battery charger can be attached directly to the receiver on some models.
2. Screw the three long antenna poles together (the shorter pole may be added if necessary for taller users). Screw on the antenna and connect its cable.
3. Put backpack and/or shoulder strap on. The pouch for the data logger should be in place around the waist strap or in the backpack.

4. Screw antenna to the attachments on the top of the backpack. Wind cord around pole, and ensure the antenna is secure. Please be aware of overhead hazards, especially if working near low-hanging power lines. Severe injury or death can result.

Basic Operation of the Pro XRS

Recording a Feature

Before beginning field use, ensure that all GPS configurations and settings are set correctly for the particular use of the Pro XRS and that an appropriate data dictionary is loaded onto the TSC1 (see Attachments 2 and 3 for typical settings). These steps outline the basic use of the GPS to document a sample position or any other defined "feature." Note that the TSC1 has both hard and soft keys that allow for its operation. The hard keys comprise all of the keys (e.g., letters and numbers) on its surface. The soft keys are the F1 through F5 hard keys. The function of these changes depending upon the context. These keys will be referred to with brackets around them (<soft-key>).

1. Turn data logger on outside in an open area. Wait for antenna to receive satellite signals. The display will read Recording Almanac, Too Few SVs, and PDOP Too High. Continue to wait until enough satellites (four) are acquired and the PDOP is below 5.0.
2. Ensure that the real-time settings are correct according to the parameters listed in Attachment 2.
3. Select **Data Collection**, and create a new rover file or open an existing file. This file should be named according to the format specified by the project GIS analyst. Note: If opening an existing file, press **<NEW>** to access the *Antenna Options* menu and *Start Feature* menu.
4. Enter the height of the antenna from the ground to the *Measurement Method* reference point shown in the *Antenna Options* menu and then press **ENTER** to bring up the *Start Feature* menu.
5. Pick the appropriate data dictionary to use with the rover file. Only one dictionary can be used with a rover file. Consult with the project GIS analyst to formulate the most appropriate data dictionary for the type of sampling you wish to perform. The data dictionary titled *Generic* contains only a comment field and is appropriate for simple navigation tasks. If using a data dictionary, make sure to become familiar with its attributes before recording information in the field.

6. Move to the location of the first feature for which you want to record the GPS position. Select the appropriate feature and press **ENTER** to begin logging. Log data points in accordance with the feature type. Point features should have at least 10 points collected at a stationary location. Line features should be collected while moving. If movement is stopped, press the **<PAUSE>** key. When movement starts again, press the **<RESUME>** key. Area features should be collected with enough points to define the outline of the area (e.g., a square building would have four single points, collected on each corner, and the **<PAUSE>** key would be used between each of the points).
7. Depending on the setup of the data dictionary, each feature may have one or more feature attributes. An attribute is used to record additional data associated with the feature. For example, the attributes assigned to a sediment sampling station could be the sample number, station ID, sampling gear, sediment color, odor, etc.
8. Use the **<PAUSE>** key while recording feature attributes to avoid too many data points being collected at one point feature. (Body movements while logging attributes for an extended time can decrease the accuracy of collection.) The **<PAUSE>** key must be used when recording attributes of a line or area feature because only one data point should be collected in a single location.
9. Once all attributes are entered and the feature data points are logged, press **ENTER** to complete and save the feature and move on to a new feature. Pressing **ESC** instead of **ENTER** will allow the user to abandon the logged feature without saving.
10. When all features in a given area have been recorded, from the *Data Collection* menu, press **ESC** to exit data capture and then press **<YES>** to close the file. Features are appended and saved to the file after each collection, so there is no need to "save" the file. When the Pro XRS is not in use, it should be turned off. If you need to come back to the same rover file later in the day, the rover file may be reopened at that time. Rover files may not be edited after 7 days from the first feature was created. Please consult the project GIS analyst for the best way to handle multi-week sampling projects.
11. At the end of each day, download the rover file to a PC using Pathfinder Office software.

Feature Collection Options

Offsets—The Pro XRS can collect a point or line feature while standing at a set distance away from the feature. This option may be necessary because of obstructions such as tree cover, buildings, or car traffic. For a point feature, measure the distance between the object you want recorded and the Pro XRS antenna. Use the compass to determine the bearing (e.g., west is 270°). The bearing is the direction the point should be moved for it to be located in the correct place (e.g., if you are due north of the feature, the bearing is south, or 180°; i.e., the position you want recorded is south of where you are standing). Estimate the inclination from the

feature to the GPS antenna (if altitude determination is critical, a clinometer should be used). The inclination is the degree angle up from the feature to the antenna (e.g., if the feature is 5° below the antenna position, enter −5°). During data capture, from within the feature, press the **<OFFSET>** button, and enter the distance, bearing, and inclination. Press **OK** to complete the feature. Note: This procedure describes an offset of a single feature. A constant offset may be applied to all features collected as well.

Nesting—While recording a line feature or an area feature, a point feature may be collected to avoid backtracking. While recording the line or area feature, press **<PAUSE>** and then **<NEST>**. The Pro XRS will prompt for collection of a new feature. Move to the feature, and collect data as for any other point feature. When the feature is complete, press **OK**. The Pro XRS is ready to resume collecting data as part of the line/area feature: press **<RESUME>**. (Remember to continue moving before pressing resume to avoid having multiple positions recorded in the same place in the line or area feature.)

Segmenting—While moving along a line feature, changing the attributes of that line may be necessary (e.g., because of a change in surface type from paved to dirt road). This change may be done without having to begin a new feature by pressing **<PAUSE>** and then ****. Change the appropriate attributes and then press **<RESUME>** to continue recording.

Repeat—This function allows the collection of a new feature with the same feature attributes as the previous feature. If features are not exactly the same, it also allows editing of the attributes.

Quickmark—Allows collection of point features while moving (e.g., from a car or a boat) by estimating the exact location. The use of this feature will not result in positionally accurate locations and is not recommended for most sampling operations.

Reviewing and Editing Features

It is possible to review or edit features collected in the field while still in the data capture mode. For example, it may be necessary to document the GPS location in the field logbook or to edit one of the feature's attributes. Without exiting data capture, press **<REVIEW>**. (If data capture is already complete, just press **<REVIEW>** and then select the appropriate rover file.) This step will display a list of data points including each feature collected. Scroll to the appropriate feature, and follow the steps below depending on the required action:

- To view the GPS location (e.g., lat/lon), press **<POS>.**
- To edit the attributes, press **ENTER**. Make any necessary edits to the attributes by scrolling through.
- To change or add an offset, press **<POS>** and then **<OFFSET>**. Make any necessary changes.
- To delete a feature collected in error, press **<DEL>**.

Navigating to an Existing Location

Waypoints

To use the Pro XRS to navigate to a previously established position, this position must be loaded into the data logger as a waypoint, present as a feature position in the data files, or generated in the field using the GPS unit. Waypoints may be entered into the TSC1 by:

- Entering coordinates manually
- Choosing previously recorded locations and importing them into the TSC1 by using Pathfinder Office
- Defining a location stored in a rover file saved to the TSC1 as a waypoint (see *Reviewing/Editing Features*, above)
- Creating a way point from the current position being shown by the operating GPS unit in the field.

Navigating

Usually you will use the *Navigation* module (accessed by pressing **MENU** followed by **Navigation**) to guide yourself to a target (waypoint or feature). You can also use the *Map* module (accessed by pressing **MENU** followed by **Map**) to:

1. Orient yourself in the area where you are working.
2. Get a general indication of the location of a feature or waypoint that you want to find.
3. Find or select features or waypoints to which you wish to navigate toward.
4. Plot a course from one place to another.
    a. While in the Map screen, the GPS cursor x shows the current position reported by the receiver and is always shown on the Map screen (Note: it may not always be within the visible part of the screen when panning or scrolling). The **<OPTIONS>** key can be used to hide or display the GPS trail (line of dots showing up to 60 previous positions), the heading showing the direction of travel, and other options on the map display.
    b. Select a feature by pressing **MENU**, Data Collection to reach the *Start Feature* screen, and then **<REVIEW>** to access all features contained in the data file. Highlight and select the desired feature by pressing the **<Target>** key, which adds a crossed flag to the feature. Reaccess the *Map* screen by selecting **MENU**, then **Map**, which will now show the highlighted feature with a crossed flag symbol on the Map screen. You can then start moving toward the feature, and the current position (shown by the x) will move closer to the target position as the user approaches.

c. There are two graphical modes of navigation with the Pro XRS in the TSC1 *Navigation* module. On both modes, text information appears on the right of the screen in the *Info* panels, which can be configured by the user. The graphical modes available are the *Directional Dial* screen or the *Road* screen, which can be toggled between using the **<Mode>** key.

d. To navigate, select a target and then a start position. Each of these positions can be features from an open data file or a waypoint. Access a list of available features or waypoints by pressing **<TARGET>** or **<START>**. Once the item has been chosen as a target, it will show the crossed flags symbol in the list. Once a target has been selected, *Distance to Go* appears at the bottom of the *Navigation* screen, which indicates the distance from the current GPS position to the target. Select a start position (not required but useful for calculating crosstrack error and other navigation information) by pressing **<START>**. A waypoint of the current GPS position can be created for use as the Start point by selecting **<CREATE>**. Once the Start position is selected, a flag symbol will appear next to the item in the list.

e. In the *Directional Dial* mode, an arrow will appear that will always point at the target. This is the bearing to go. (Note: You need to be moving for this to be accurate, as it will lock if you are moving too slowly or have stopped.) The triangle at the top represents the direction that you are going or heading. This triangle never moves, but by changing directions, you can line up the arrow with the triangle. When the two are aligned, you are heading in the direction of the target. When you are close to the target, a bull's-eye (two concentric circles) will appear at the edge of the screen. This is warning you that the unit will be switching to the close up screen. A proximity alarm will sound and the directional arrow will be replaced by the bull's-eye on the close up screen. Your current position will be shown by an x and the target by the bull's-eye. Move so that the x is in the same location as the bull's-eye.

f. In the *Road* mode, navigate by walking down a road. Your position is shown by a stick figure and is always positioned in the center of the screen. The target (crossed flags) shows the point to which you are navigating toward. Your heading is shown by the top center of the screen and the bearing to go is shown by the direction of the road, which will rotate as you change your heading. Change your heading until the road is pointing at the top of the screen (*Target* is also at the top of the screen) and the edges are parallel to the sides of the screen. As you move toward the target the screen zooms in, so the road appears to get wider.

Downloading Rover Files

Upon returning to the office, download all rover files from the TSC1 to a PC for post-processing. You will need the Trimble Pathfinder software installed on your computer. If you

are not using a field laptop that already has the program installed, contact your project GIS analyst for instructions on how to install the software.

Connect the TSC1 to your computer using the appropriate cables. In addition to the "pigtail" cable, you will also need a null modem (a 9-pin female-to-female cable) to plug into a PC serial port. Once connected, power up the TSC1 unit and navigate to *MENU>File Manager>File Transfer*. Then, open the Pathfinder software and navigate to the *Utilities>Data Transfer…* window from the menu bar. Select **GIS Datalogger** on COM1 (for most computer systems), and press the green **Connect** button. Download files from the TSC1 by selecting the **Receive** tab and choosing the data file type from the *Add* pulldown menu (Figure 1).

After downloading, remove all rover files and waypoints from the TSC1 to conserve memory. Rover files may be deleted from the *File Manager* menu as follows:

1. Select **MENU>File Manager>Delete File(s)**
2. Select the rover file to be deleted, and press **<ENTER>**
3. Confirm the deletion of this file by pressing **<YES>**.

Delete data dictionaries in the same manner by selecting **Data Dictionaries** from the *File Manager* menu. Delete waypoints by selecting **Utilities** from the *Main* menu and then by selecting **Waypoints,** followed by **<DEL>**.

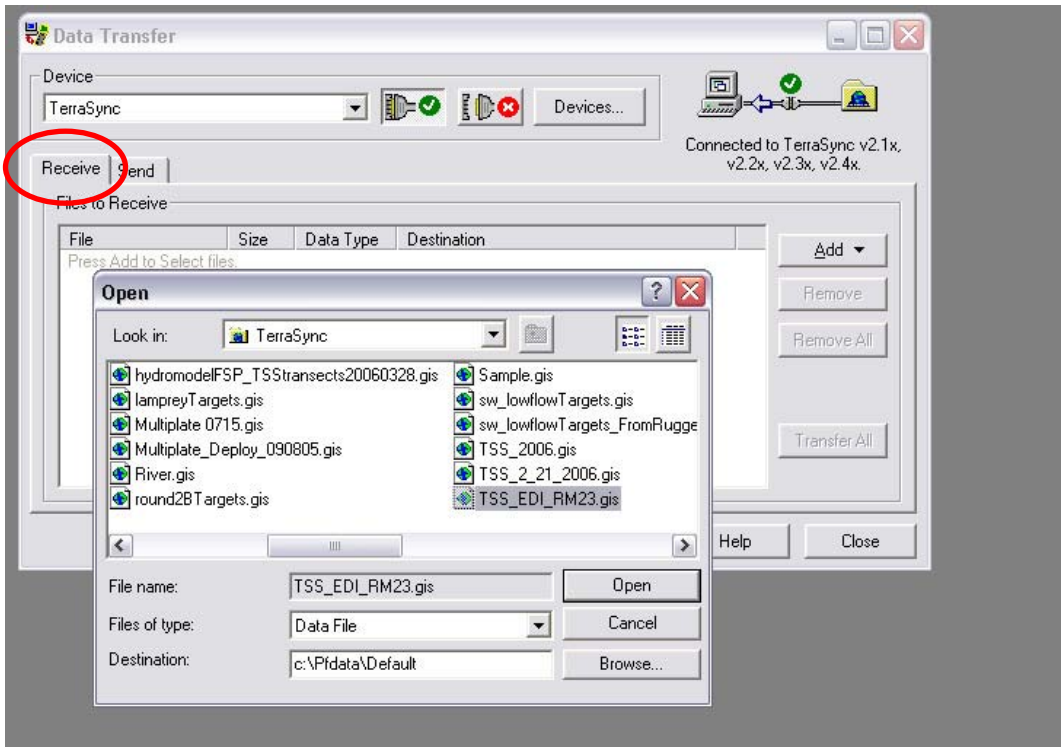

Figure 1. Transferring File from Terrasync

ATTACHMENT 2
TSC1 SETTINGS

The following are lists of menus that can be accessed through the TSC1 keypad. Please ensure that settings are correct before proceeding. Do not make changes to the settings unless necessary. Each menu will list all available subheadings, the correct setting, and the available **<soft-keys>** to access additional menus. Comments are included only where necessary.

GPS Rover Options

To access this menu, select **Configuration** from the main menu and then select **GPS Rover Options**. The table below lists logging options and settings.

Logging Options	Setting	Comment
Logging intervals		
Point feature	1s	
Line/area feature	2s–5s	depending upon speed of movement
Not in feature	None	
Velocity	None	
Confirm end feature	No	
Minimum pos	10	
Carrier Mode	Off	
Carrier phase min. time	10 minutes	
Dynamics code	Land	May be changed to sea or air, as appropriate
Audible click	Yes	
Log DOP data	Yes	
Log PPRT data	Yes	
Log QA/QC data	Yes	
Allow GPS update	Warn First	
Warning Distance	Any	
Position Mode Manual	3D	
Elevation Mask	15°	Should not go below 13° (accuracy decreases)
SNR Mask	6.0	Can raise to 7 if multi-path filtering is poor
PDOP Mask	5.0	Can be raised up to 8; reduces accuracy
PDOP Switch	6.0	

Real-Time Input Options

Access this menu from the GPS Rover *Options* menu by selecting **Real-Time Input**. The table below shows options and settings for real-time input.

Options	Setting	Comment
Preferred Correction Source		
	Choice 1	Integrated Beacon
	Choice 2	Integrated WAAS
	Choice 3	Use uncorrected GPS
Correction Age Limit	20s	

Antenna Options

Access this menu from the GPS rover *Options* menu by selecting **Antenna Options**. The table below shows antenna options and settings.

Option	Setting	Comment
Height	6 ft	Enter correct user antenna height using measurement method indicated below
Measure	Uncorrected	
Type	Integrated GPS/Beacon/Satellite	
Confirm	Per file	Can be changed to “Per feature” if antenna height varies and elevation is critical
Part Number	33580-50	Auto selected based on TYPE selected
Measurement	Bottom of Antenna	
Method	Mount	

ATTACHMENT 3
ADDITIONAL SETTINGS FOR THE TSC1

Additional TSC1 settings can be found in the *Configuration* menu. Items of particular importance are indicated in italics.

Configuration

This menu can be accessed by selecting **Configuration** from the main menu. The table below lists options and descriptions for the *Configuration* menu.

Options	Description
GPS base station options	For using a land base station or beacon for real time corrections
NMEA/TSIP output	Consult manual
Coordinate system	Changes coordinate system among latitude/longitude, UTM, and other coordinate systems. System can be converted, if necessary, after data capture by using Pathfinder Office software.
Map Display options	Change layers, scale, background files and items shown on the TSC1 screen during data collection
Navigation options	Changes Navigation parameters
Units and display	Changes various units, for example: length (e.g., feet, meters), altitude reference (e.g., MSL), *North reference* (i.e., true or magnetic). Units can be converted, if necessary, after data capture by using Pathfinder Office software.
Time and date	Changes to *local time*, 24-hour clock, date format, and other options
Quickmarks	Set-up parameters for use with Quickmarks.
Constant offset	Set-up parameters for use with a constant offset.
External sensors	Connections with external sensors.
Hardware (TSC1)	TSC1 settings such as beep volume, contrast, *internal and external battery status*, software version, free space.

Contrast and Backlighting

The TSC1 display can be viewed in various light settings. Press **FUNC,** then **L** to turn on the display backlight for viewing in dim lighting. Adjust the contrast by pressing **FUNC**, then **E** or **F**.

ATTACHMENT 4
PRE-SAMPLING ACTIVITIES BEFORE USE OF THE PRO XRS

Determination of Optimal Satellite-Use Time

Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoiding these time intervals permits the operator to maintain better positioning accuracy.

ATTACHMENT 5
MANAGING GPS DATA FROM TERRASYNC—A TUTORIAL

Currently, positional data collected in the field is most often done with a Trimble GPS unit (usually rented) interfaced with a laptop via Trimble's Terrasync software. The Terrasync software sometimes exhibits quirks that interfere with the smooth operation of data collection in otherwise stressful field conditions. This tutorial is meant to supplement the Terrasync software documentation and serve as a guide to field personnel to help them retrieve and collect geographic data as efficiently as possible with existing software.

Scope

This document is intended to be a reference for procedures involving the following:

- Fixing files that are more than 7 days old so that they can be updated
- Adding features in GPS Pathfinder software (companion to Terrasync) and then importing them as base files in Terrasync..

This document is not intended to be a comprehensive manual for using Terrasync or Pathfinder software. It is assumed that the reader has received at least some training on how to use the basic features of Terrasync and is competent at using MS Windows.

The Basics

GPS data collection currently relies on two pieces of complementary software:

- Terrasync—the interface for GPS navigation and data collection.
- Pathfinder Office—a multiuse piece of software that acts as a conduit between GIS data files (shape files) and Terrasync GPS files. Pathfinder can also be used as a simple map editor.

Installing the Correct Versions of Terrasync and Pathfinder

Important Note: This tutorial uses Pathfinder Office v. 3.00 and Terrasync v. 2.50. It is very important to use the proper versions of this software to avoid compatibility issues. These software versions should be included in the same folder as this tutorial, or can be obtained from GIS staff.

http://www.trimble.com/terrasync_ts.asp?Nav=Collection-4576

Key code for TerraSync
499043-00110-05273-EDD049BC

Pathfinder v.3.00
001533-00300-04152-0ee4d11f

Initial Setup of Terrasync/Pathfinder

Certain settings and configuration setups are needed before Pathfinder can talk to Terrasync. Whether you are installing this software for the first time or have an existing installation, check to make sure that these settings are in place.

1. Open Pathfinder Office and go to the *Utilities>Data Transfer...* menu. A dialog box should appear. This is the interface for communicating with Terrasync.
2. Click the **Devices** button, and then **New…** (Figure 1).
3. Click on **GIS Folder**.
4. Browse to the Terrasync data folder on your computer, which in most cases will be *C:\My Documents\TerraSync*.
5. In the next box, *Type* will be **Terrasync**, and *Version* will be **v. 2.1x, v.2.2x, v.2.3x,** and **v2.4x**.
6. At the prompt for a name that will display in the device list, enter **Terrasync**.
7. Go back to the Data Transfer dialog box, select **Terrasync** from the dropdown menu, press the **Connect** icon, and look for a green check mark indicating success.

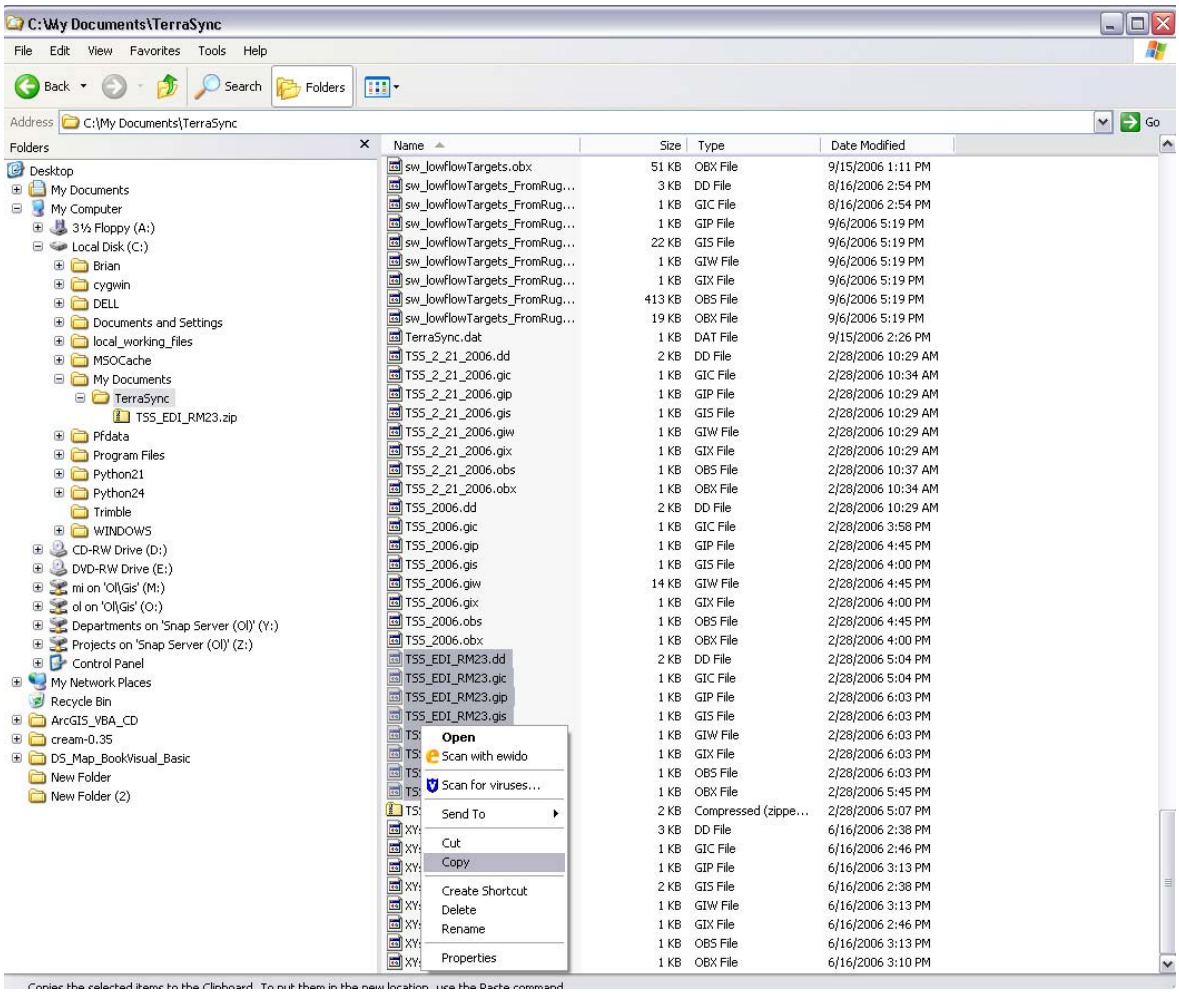

Figure 2. Selecting Files To Copy to a Different Directory

If this procedure does not work for you, you may have the wrong version of Pathfinder. For some unknown reason, with each version upgrade of Pathfinder, connectivity to older versions of Terrasync is lost. You can check what version of Pathfinder you have installed by going to the *Help>About GPS Pathfinder Office...* menu. To find out what version of Terrasync you have, go to *C:\Program Files\TerraSync*, right-click on **Terrasync.exe**, and choose the **Version** tab.

Handling Expired Files in Terrasync

One of the most common problems that field personnel will have to deal with is the 1-week expiration date when trying to collect data with Terrasync. This is a built-in function of Terrasync, and there is no simple way to work around it. The following instructions will guide you through the process to make the files usable. See Figure 3.

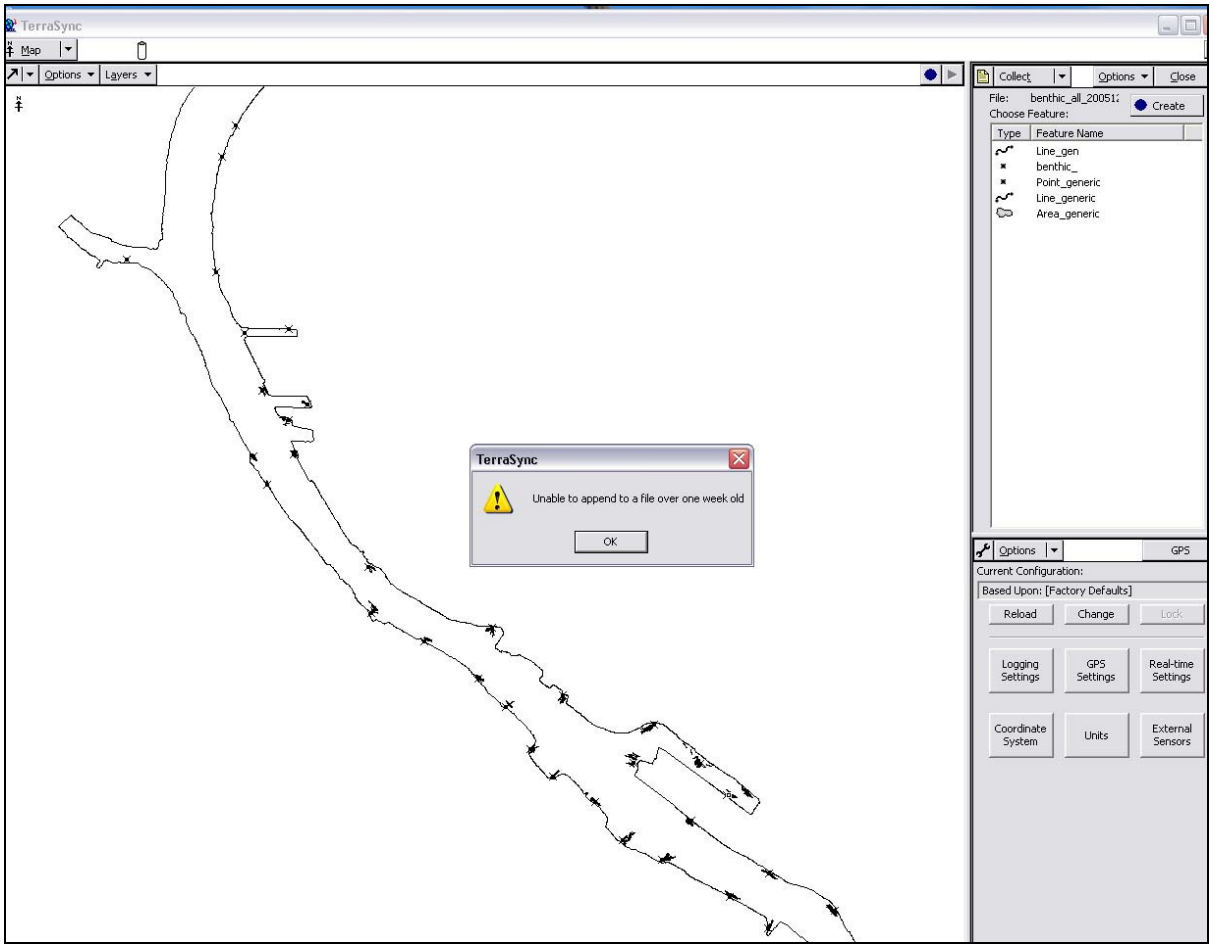

Figure 3. Notice That Terrasync File Older Than 1 Week Will Not Allow User To Collect Features (time begins to elapse when first feature is collected in the field, not when file is created)

Two options are available, depending on your needs. If you do not need to see the previously logged locations and need only to see the targets, use the original files provided by GIS staff (Option 1). If you need to see previously occupied locations in order to make decisions about where to go next, then transfer the file to Pathfinder and back again (Option 2).

Option 1: Move and replace logged files with original targets.

At the beginning of the field effort, you should receive a set of files with the target locations, most likely in a zip archive (.zip file extension). There will be six to eight files with the same name but with different extensions (Figure 4). These files will have to go into the *C:\My Documents\TerraSync* folder in order to be available to Terrasync.

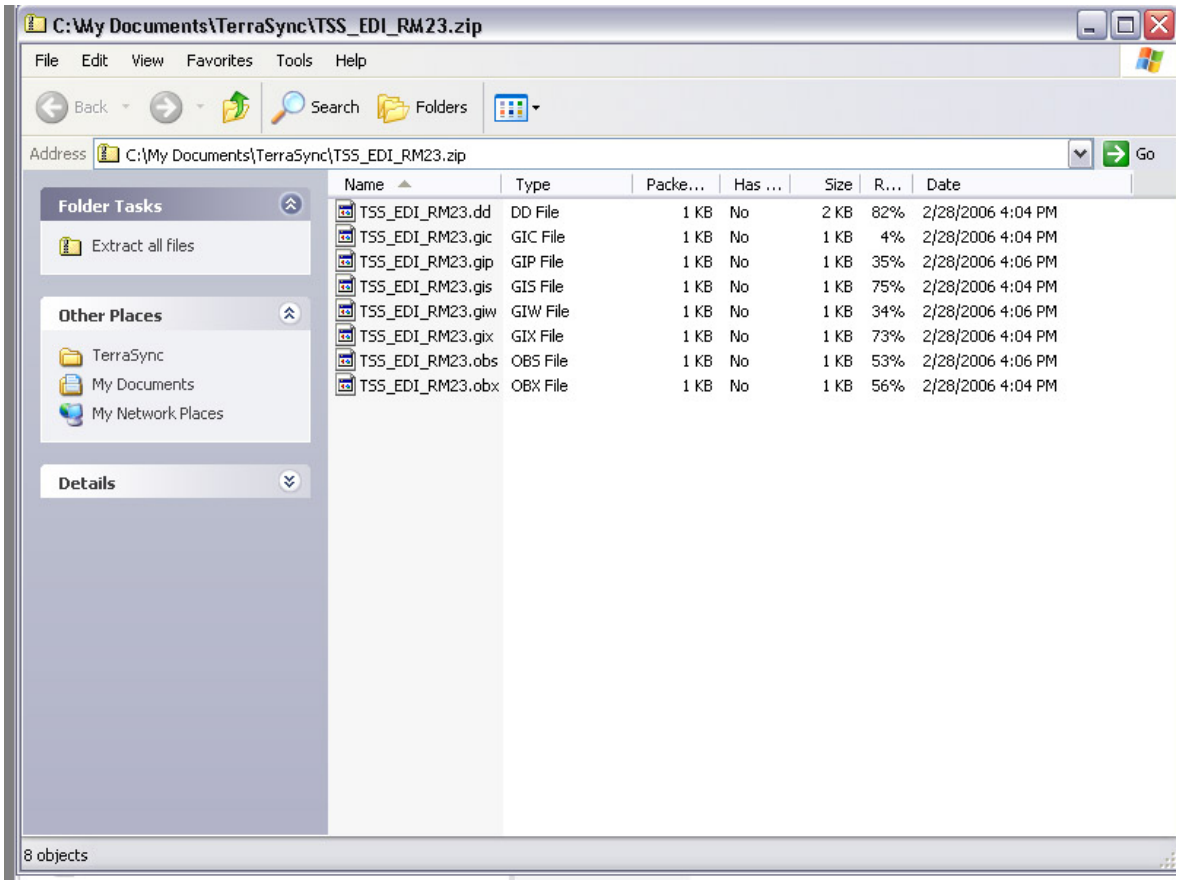

Figure 4. Example of File Set To Be Unzipped into the Terrasync Folder

After you unzip these files to Terrasync, keep this zip archive around in an easy-to-find place, such as your computer desktop, because the 1-week clock does not start until you begin collecting your first point in the field. You can use this unadulterated file again, as long as you make a copy of the work you did the previous week. The detailed steps are as follows:

1. Make sure you have the original files with the target locations available in a handy place. This will probably be the original zip archive. Also, be sure to close Terrasync while performing this process.
2. Navigate to C:\My Documents\TerraSync\ in Windows Explorer. Locate the files that you have been using the previous week. Note: It is crucial to get all of the small files associated with the data set. While it is useful to sort the files by date modified, you can miss some of the small files—it is highly recommended that you sort the files alphabetically.

3. Copy all of these files to a different directory, preferably one that is named appropriately to reflect the data and time period that you were collecting. For example: C:\Documents and Settings\bpointer\Desktop\lampreyTargets_20060925. These files contain the data you have collected the previous week and should be backed up and/or emailed to the appropriate project manager or GIS staff.
4. You can now safely replace the files you just copied with the ones from the original zip file. Right-click the zip archive, and click Extract All. When prompted to Select a folder to extract files to, browse to C:\My Documents\TerraSync. (Figure 5). If prompted about replacing existing files, select Yes to All. Note: It is crucial to make copies of the files first (see Step 3 above)—otherwise, you may lose the data.
5. You should now be able to open the file in Terrasync and begin logging as normal.

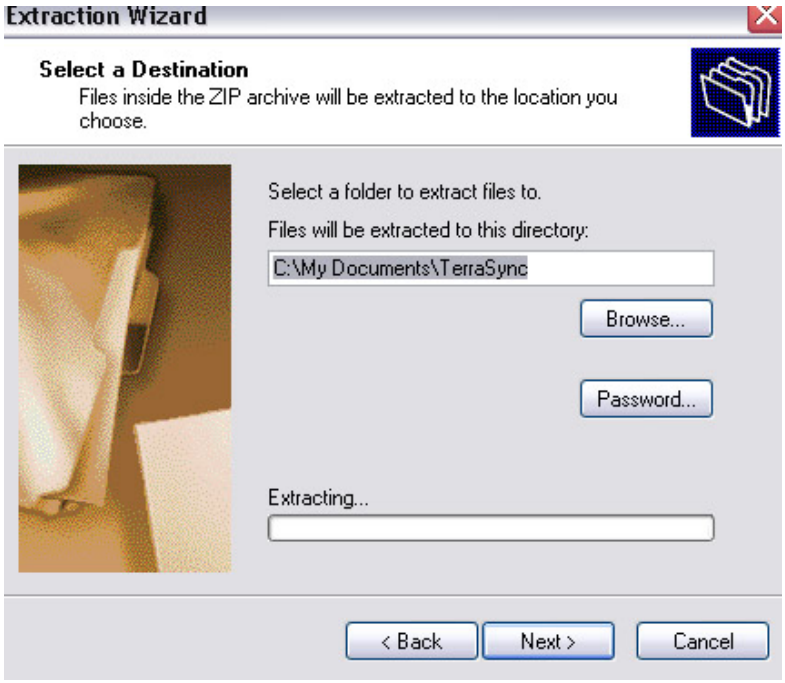

Figure 5. Extract (or copy) Original Target Files into the Terrasync Directory

Option 2: Transfer files back and forth from Terrasync.

If you need to be able to see the previously occupied positions from last week while positioning this week, you need to use Pathfinder to reset the file. This process will essentially combine the targets and actuals from last week into one file. However, this method has its drawbacks; once converted, the actuals from last week will not be able to be corrected, so a backup procedure similar to the one in the previous option should be carried out to maintain data integrity.

The steps for file transfer are as follows:

1. For good data management, back up the data files from the previous week using the procedure laid out in steps 1 through 3 in Option 1 above.
2. Close Terrasync and open up Pathfinder Office.
3. Go to the Utilities>Data Transfer menu or just click the icon on the left (Figure 6).
4. Ensure that the device listed is Terrasync. If not, follow the initial setup instructions at the beginning of this document. Most of the computers used for GPS logging are already setup for this.
5. There are two tabs, Receive and Send. Make sure that Receive is selected and then go to Add>Data File. Select the file(s) that you are using and select Open. The file should now be in the Files to Receive box. Click Transfer All and wait for the transfer to take place. If you have made the recommended backups, it is fine to replace any files.
6. Now select the Send tab (Figure 7), and go to Add>Data File. Select the file you just transferred (it will have the same name as the Terrasync file) and click Open. Now click Transfer All to move the file back to Terrasync.

   By transferring the file back and forth from Terrasync to Pathfinder, you have "reset the clock" and can now update the file for an additional 7 days. This file will have your targets and actual positions from the last week, so it is important to be aware of the features you are selecting for navigation.

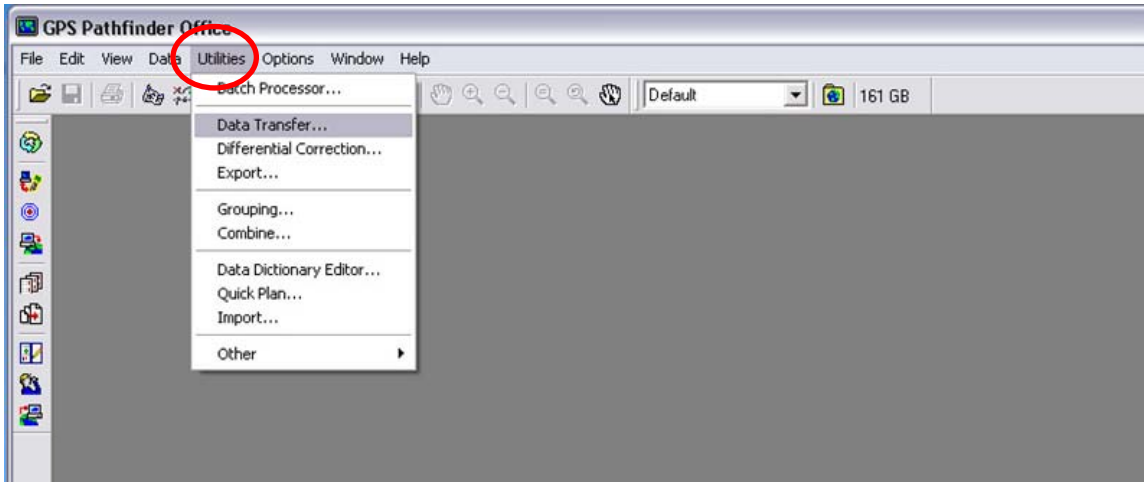

Figure 6. Data Transfer Menu

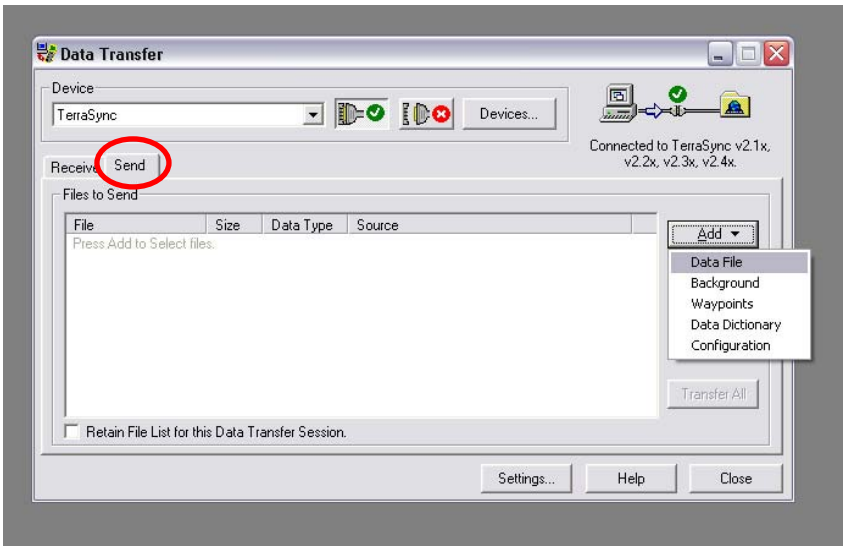

Figure 7. Sending Data File

STANDARD OPERATING PROCEDURE (SOP) SL-01

DECONTAMINATION OF SOIL SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either organic or inorganic materials. To prevent potential cross contamination of samples, all reusable soil sampling and processing equipment is decontaminated before each use. At the sample collection site, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All soil sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the site-specific health and safety plan (HSP).

Sampling equipment may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment (e.g., hand auger, split-spoon sampler) used for both analyte groups should follow the order of a detergent wash, site water rinse, organic solvent rinses, and final site water rinse. Sample processing equipment (e.g., bowls, spoons) is rinsed with distilled/deionized water instead of with site water.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following:

- Steam cleaner and collection basin (if required)
- 55-gal, Department of Transportation (DOT)-approved drums (if required)
- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or site water (i.e., potable water)
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial nonphosphate detergent
- Pesticide-grade ethanol and hexane (consult project-specific field sampling plan [FSP], as the solvents may vary by U.S. Environmental Protection Agency [EPA] region or state)
- 10 percent diluted nitric acid or hydrochloric acid (reagent grade) for inorganic contaminants (if required; see project-specific FSP)
- Baking soda (if required)
- Long handled, hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Personal protective equipment as specified in the HSP.

PROCEDURES

Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not one of the sample analytes. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is slightly more effective than other solvents, its use is discouraged because of its potential toxicity to sampling personnel. Always follow the procedures listed in the site-specific HSP when decontaminating sampling equipment (e.g., always stand upwind when using volatile solvents, wear appropriate gloves and safety glasses or goggles). Containerize all decontamination fluids for proper disposal, following procedures listed in the FSP.

The specific procedures for decontaminating soil sampling equipment and soil compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove visible soil. This step should be performed onsite for all equipment. After removing visible solids, set aside sampling equipment that does not need to be used again that day and see that it is thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles, using a back-and-forth motion. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double rinse the equipment with tap or site water and set upright on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface. If acid and solvent rinses are not required by the FSP, skip to step 8.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle using a 10 percent acid solution. Double-rinse equipment with tap or site water and set right-side-up on a stable surface to drain. If solvent rinses are not required by the FSP, skip to step 8.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). These solvents act primarily as a drying agent by scavenging water from the equipment surface and carrying it away, but they also work as a solvent for some organic contamination. Hand-augers must be held over the waste container and turned slowly so the stream of solvent contacts the entire surface. The sample apparatus may be turned on its side, and if applicable, opened to be washed more effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.
7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container, which may need to be equipped with a funnel. Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the

equipment was not thoroughly rinsed with ethanol or that the ethanol that was purchased was not free of water. When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the ethanol and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.

8. Do a final rinse with site water for the sampling equipment (i.e., hand-auger) and distilled/deionized water for the processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collection or processing equipment is precleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.
10. After decontaminating all of the sampling equipment, dispose of the disposable gloves and used foil per the procedures listed in the project-specific FSP. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles for disposal at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda or containerized and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Metals and Conventional Parameters Only

The specific procedures for decontaminating soil sampling equipment and soil processing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove the visible soil. Perform this step onsite for all equipment. Set aside any pieces that do not need to be used again that day see that they are thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.

3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double-rinse the equipment with tap or site water (if an acid rinse is required) or with distilled/deionized water (if no acid rinse) and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle containing a 10 percent acid solution. Double-rinse equipment with distilled/deionized water and set right-side-up on a stable surface to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.
7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Drill Rig or Test Pit Sampling Equipment

1. Decontaminate sampling equipment before use, between samples and stations, and upon completion of sampling operations.
2. Equipment used during drilling/test pit operations should be decontaminated in the Exclusion Zone prior to transport to the Support Zone (refer to site-specific HSP).
3. If the steam-cleaning location is in an area outside of the Exclusion Zone, remove loose soil on the drill rig, augers, drill pipe, and rods, and other large equipment at the drill site, then move the equipment directly to the steam-cleaning decontamination area for more thorough cleaning.
4. To decontaminate a drill rig or backhoe, pressure wash with a steam cleaner using potable water rinse upon mobilization, between drilling locations, and upon demobilization. Cleaning water can generally be allowed to drain directly on the ground near the station (refer to the FSP).

5. To decontaminate auger, drill rods, and other down-hole tools, pressure wash with a steam cleaner and potable water rinse upon mobilization, between drilling locations, and upon demobilization. All decontamination fluids are to be containerized for proper disposal.
6. To decontaminate split-spoon and hand-auger samplers, follow the decontamination procedures listed above (the selected decontamination procedures is dependent upon analyte list provided in the project-specific FSP). To the extent possible, allow to air dry prior to sampling. If the split-spoon is not used immediately, wrap it in aluminum foil. All decontamination fluids are to be containerized for proper disposal.

STANDARD OPERATING PROCEDURE (SOP) SL-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SOILS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates (MS/MSDs), equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material) for soil samples. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if volatile organic compounds (VOCs) are to be analyzed. Definitions of all potential quality control samples are described below.

As part of the quality assurance and quality control (QA/QC) program, all field quality control samples will be sent to the laboratories blind. To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers that are required to complete the field quality control sample for the applicable analyte list must be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should only be recorded in the field logbook or field sampling forms. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent this from happening, select and mark regular samples on the chain-of-custody/sampling analysis request (COC) form or instruct the laboratory to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Prepare field quality control samples at least once per sampling event, and prepare certain types more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality

control sample per 20 is indicated and 28 samples are collected, prepare 2 quality control samples. The method of preparation and frequency of field quality control samples required for soil sampling activities are described below. These protocols must be followed, unless different frequency requirements are listed in the FSP and QAPP.

For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if VOCs are to be analyzed. The following table lists the possible quality control sample types and suggested frequencies for soil sampling programs (not all types of quality control samples will always be collected; see project-specific FSP and QAPP for actual quality control samples that need to be collected for a particular sampling event). A detailed explanation of each type of quality control sample with the required preparation follows.

Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1:20 thereafter
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler
Environmental (transfer) blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Collect field duplicate (or split) samples to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Prepare field duplicates by collecting two aliquots for the sample and submitting them for analysis as separate samples. Collect field duplicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of field duplicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Prepare field replicates by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Collect field replicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The project QA/QC coordinator will determine the actual number of field replicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The MS/MSD analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume that may be needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated soil stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of extra bottles collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Use equipment rinsate blanks to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Prepare equipment rinsate

blanks by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, then transferring the water to the appropriate sample containers and adding any necessary preservatives. Prepare equipment rinsate blanks for all inorganic, organic, and sometimes conventional analytes at least once per sampling event per the type of sampling equipment used. The project QA/QC coordinator will determine the actual number of equipment rinsate blanks prepared during an event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Submit bottle blanks along with soil samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, submit one bottle blank per lot of prepared bottles for analysis. If more than one type of bottle will be used in the sampling (e.g., HDPE or glass), then submit a bottle blank for each type of bottle and preservative. The project QA/QC coordinator will determine the actual number of bottle blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Use trip blanks to help identify whether contaminants may have been introduced during shipment of the soil samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two 40 mL VOC vials and tightly closing the lids. Invert each vial and tap lightly to determine if air bubbles exist. There should be no air bubbles in the VOC trip blank vials. If air bubbles are present, then note this information in the field logbook.

Transport the trip blanks unopened to and from the field in the cooler with the VOC samples. Label the trip blank and place it inside the cooler that contains newly collected VOC samples; it must remain in the cooler at all times. A trip blank must accompany samples at all times in the field. Send one trip blank (consisting of a pair of VOC vials) with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

The laboratory will use temperature blanks to verify the temperature of the samples upon receipt at the testing laboratory. The testing laboratory will prepare temperature blanks by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank must be included with each sample cooler shipped to the testing laboratory.

ENVIRONMENTAL BLANKS

Prepare the environmental (i.e., transfer) blank in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If you use unpreserved bottles, then you must add the appropriate preservative (e.g., for metals samples, use a 10 percent nitric acid solution to bring sample pH to 2 or less), if required. Collect environmental blanks at a minimum frequency of 1 in 20 samples. The project QA/QC coordinator will determine the actual number of environmental blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water and then seal. Note the location from which the environmental blank was collected along with atmospheric conditions at the time of its collection in the field logbook. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

Reference materials (i.e., a standard reference material, a certified reference material, or other reference material are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. Reference materials have undergone multilaboratory analyses using a standard method which provides certified concentrations. When available for a specific analyte, Reference material samples provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several reference materials may be required to cover all analytical parameters. For all analytes where available, one reference material will be analyzed at a frequency of one per 50 samples. The project QA/QC coordinator will determine the actual number of reference materials analyzed during a project on a case-by-case basis (consult the project-specific FSP

and QAPP, as the requirements on frequency of reference material analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SL-04

FIELD CLASSIFICATION OF SOIL

SCOPE AND APPLICATION

This SOP establishes the minimum information that must be recorded in the field to adequately document surface soil sampling and soil borehole advancement activities performed during field exploration. The surface soil sampling or borehole log form must be filled out completely for each station.

This SOP presents the field classification of soils to be used by Integral field staff. In general, Integral has adopted the procedures provided in American Society for Testing and Materials (ASTM) Method D-2488-00, Standard Practice for Description and Identification of Soils (attached). ASTM D-2488-00 uses the Unified Soil Classification (USC) system for naming soils. Field personnel are encouraged to study these procedures prior to initiation of fieldwork.

Soil descriptions should be precise and comprehensive without being verbose. The overall impression of the soil should not be distorted by excessive emphasis on minor constituents. In general, the similarities of consecutive soil samples should be emphasized and minor differences de-emphasized. These descriptions will be used to interpret potential contaminant transport properties, rather than interpret the exact mineralogy or tectonic environment. We are primarily interested in engineering and geochemical properties of the soil.

Soil descriptions should be provided on the surface soil field collection form or in the soil description column of the Integral's soil boring log for each sample collected. If there is no difference between consecutive soil samples, subsequent descriptions can be noted as "same as above" or minor changes such as "increasing sand" or "becomes dark brown" can be added.

The format and order of soil descriptions should be as follows:

- Group symbol (in the Unified Symbol column)
- USC name (should be identical to the ASTM D-2488-00 Group Name with the appropriate modifiers)
- Minor components
- Color
- Moisture
- Additional descriptions.

EQUIPMENT AND REAGENTS REQUIRED

- Surface soil field collection form or borehole log form (see SOP SL-06, *Logging of Soil Boreholes*)
- Munsell® soil color chart.

PROCEDURES

The USC is an engineering properties system that uses grain size to classify soils. The first major distinction is between fine-grained soils (more than 50 percent passing the No. 200 sieve [75 μm/0.0029 in.]) and coarse-grained soils (more than 50 percent retained by the No. 200 sieve). Small No. 200 sieves are necessary to classify soils near the cutoff size.

1. Fine-grained soils are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-00. If these tests are used, include the results in the soil description. If these materials are encountered, perform at least one complete round of field tests for a site, preferably at the beginning of the field investigation. The modifiers "fat" and "lean" are used by ASTM to describe soils of high and low plasticity. The soil group symbols (e.g., CL, MH) already indicate plasticity characteristics, and these modifiers are not necessary in the description. Soils with high plasticity can be emphasized by describing them as "silty CLAY with high plasticity." Plasticity, for example, is an important descriptor because it is often used to interpret whether an ML soil is acting as either a leaky or a competent aquitard. For example, an ML soil can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.
2. Coarse-grained soils are classified as either predominantly gravel or sand, with the No. 4 sieve (4.75 mm/0.19 in.) being the division. Use modifiers to describe the relative amounts of fine-grained soil, as noted below:

Description	Percent Fines	Group Symbol
Gravel (sand)	<5 percent	GW, GP (SW, SP)
Gravel (sand) with silt (clay)	5–15 percent	Hyphenated names
Silty (clayey) gravel (sand)	>15 percent	GM, GC (SM, SC)

The gradation of a coarse-grained soil is included in the specific soil name (e.g., fine to medium SAND with silt). Estimating the percent of size ranges following the group name is encouraged for mixtures of silt sand and gravel. Use of the modifiers "poorly graded" or "well graded" is not necessary, as they are indicated by the group symbol.

Show a borderline classification with a slash (e.g., GM/SM). Use this symbol when the soil cannot be distinctly placed in either soil group. Also use a borderline symbol when describing interbedded soils of two or more soil group names when the thickness of the beds are approximately equal, such as "interbedded lenses and layers of fine sand and silt." Do not use a borderline symbol indiscriminately. Make every effort to place the soil into a single group. (One very helpful addition to the soil log form description is the percentage of silt/sand/gravel. Even if the geologist did not have sufficient time to properly define the soil, this percentage breakdown allows classification at a later date).

3. Precede minor components, such as cobbles, roots, and construction debris with the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). Use the word "occasional" to describe random particles of a larger size than the general soil matrix (i.e., occasional cobbles, occasional brick fragments). The term "with" indicates definite characteristics regarding the percentage of secondary particle size in the soil name. It is not to be used to describe minor components. If a nonsoil component exceeds 50 percent of an interval, state it in place of the group name.
4. Give the basic color of a soil, such as brown, gray, or red. Modify the color term with adjectives such as light, dark, or mottled, as appropriate. Especially note staining or mottling. This information, for example, may be useful to establish water table fluctuations or contamination in boreholes. The Munsell® soil color chart designation is the Integral color standard. These charts are readily available and offer a high degree of consistency in descriptions between geologists.
5. Define the degree of moisture present in the soil as dry, moist, or wet. Moisture content can be estimated from the criteria listed in Table 3 of ASTM D-2488-00.
6. If observed, note such features as discontinuities, inclusions, joints, fissures, slickensides, bedding, laminations, root holes, and major mineralogical components. Note anything unusual. Additional soil descriptions may be made at the discretion of the project manager or as the field conditions warrant. The surface soil field collection and soil boring log forms list some optional descriptions, as does Table 13 of the ASTM standard. The reader is referred to the ASTM standard for procedures of these descriptions.

The contact between two soil types must be clearly marked on the surface soil field collection or soil boring log forms. If the contact is obvious and sharp, draw it in with a straight line. If

it is gradational, use a slanted line over the interval. In the case where it is unclear, use a dashed line over the most likely interval.

For drilling activities, the field geologist, who has the advantage of watching the drilling rate and cuttings removal and can talk with the driller in real time, has a much better chance of interpreting the interval than someone in the office.

ATTACHMENT 1. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-µm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols.*

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula*.

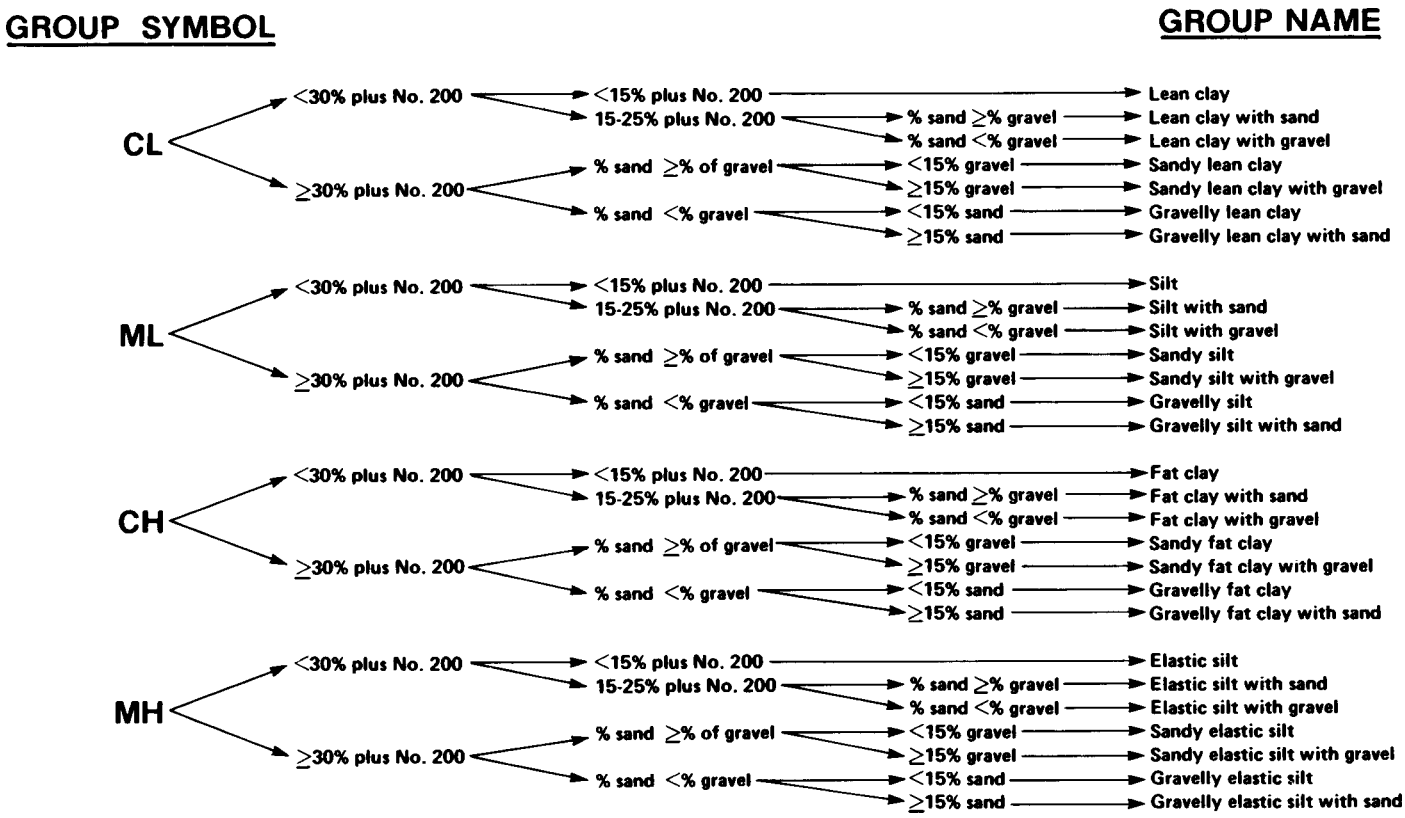
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens*.

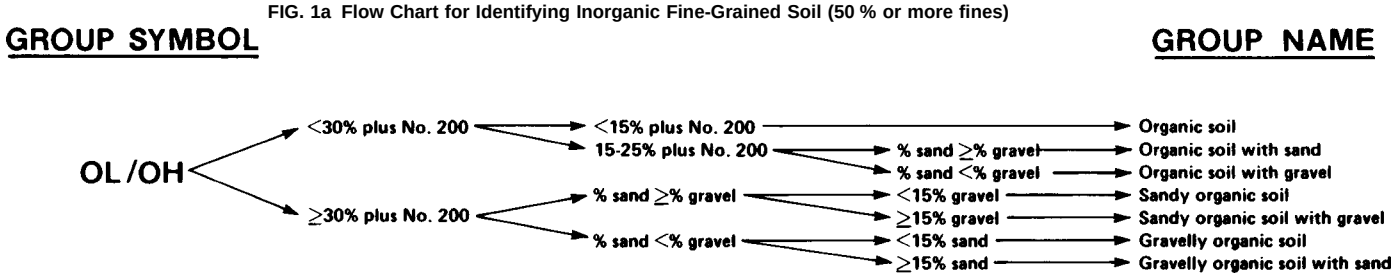
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

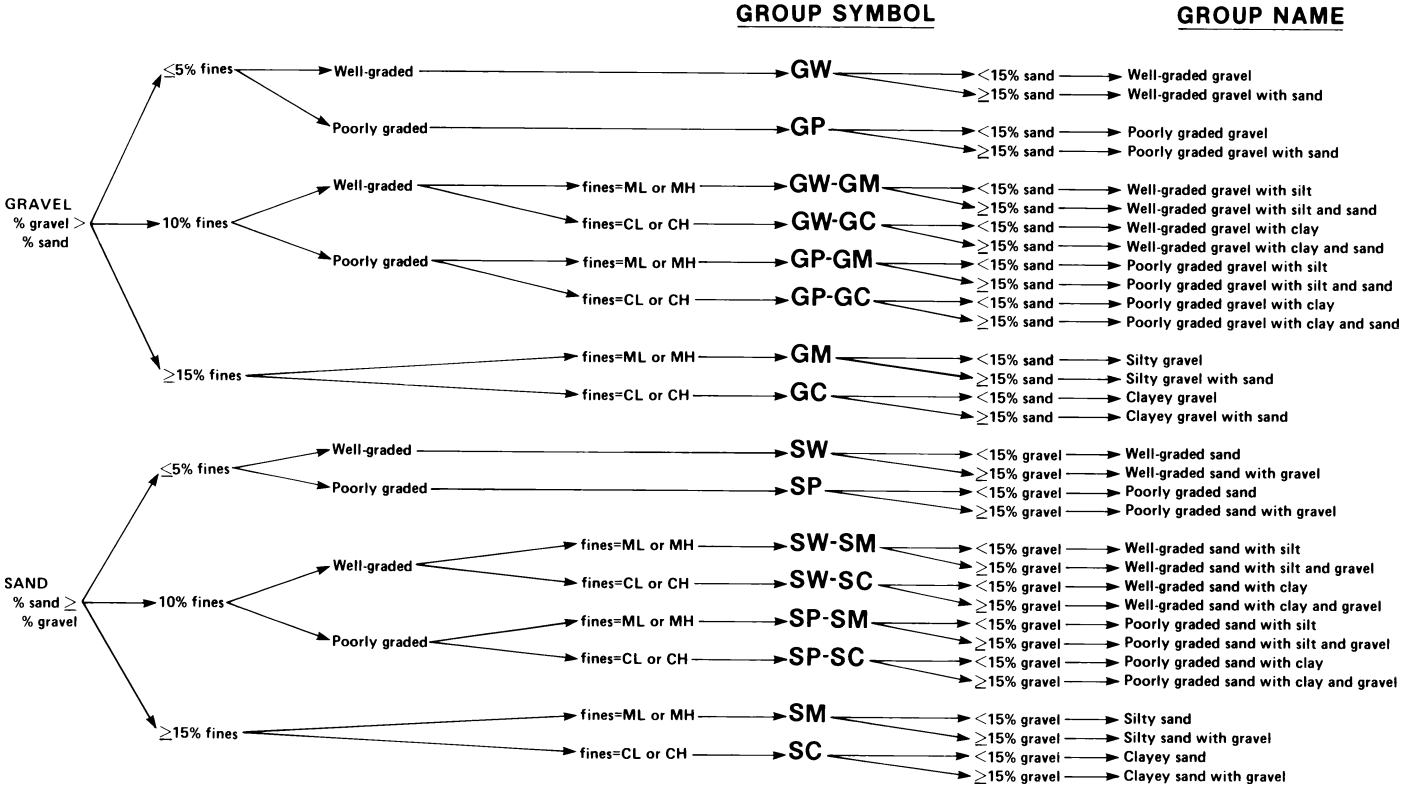
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

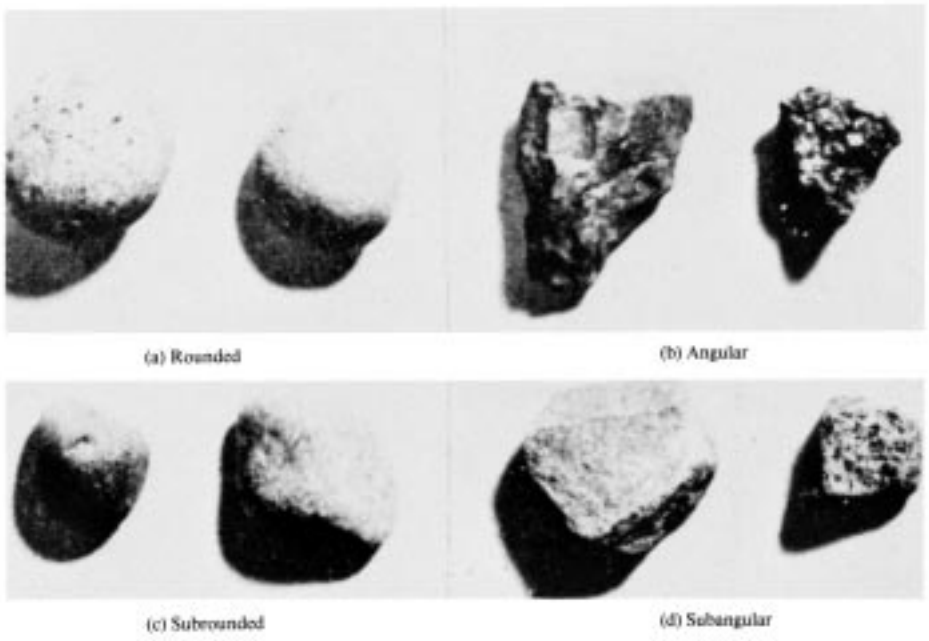


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

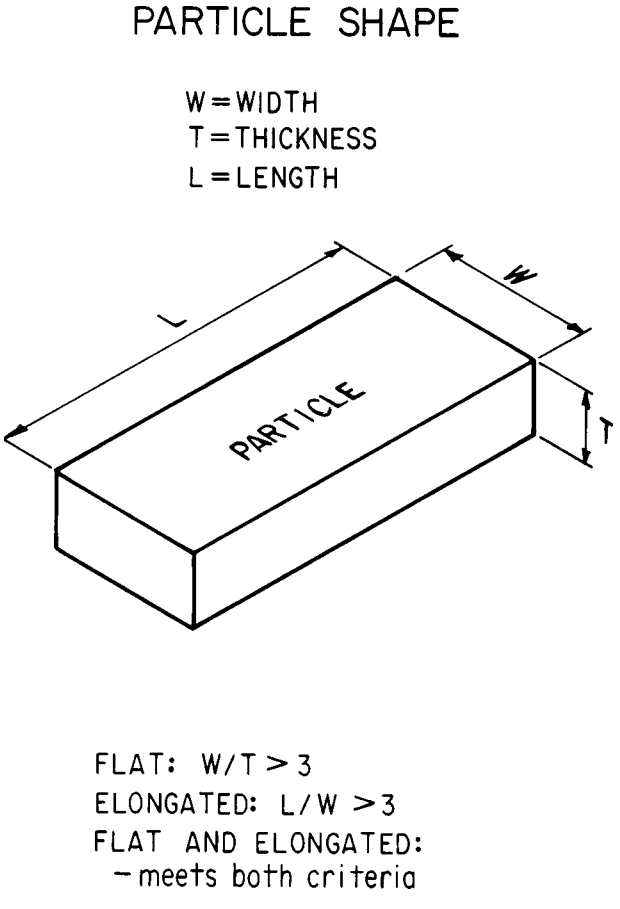


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as “Sandy Lean Clay (CL)”; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; “Poorly Graded Sand with Silt (SP-SM)”; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken

shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993$^{\epsilon 1}$) that may impact the use of this standard.

(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

STANDARD OPERATING PROCEDURE (SOP) SL-05

SURFACE SOIL SAMPLING

SCOPE AND APPLICATION

This SOP defines and standardizes the collection of surface soil samples (e.g., 0 to 12 in. below ground surface). Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

The procedures listed below may be modified in the field upon the agreement of the lead site sampler and field personnel, based on field and site conditions, after appropriate annotations have been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral's SOP on methanol preservation of soil samples. Record all pertinent information on Integral's surface soil sampling field data form or field logbook.

EQUIPMENT AND SUPPLIES REQUIRED

- Decontaminated sampling tool (stainless-steel shovel, scoop, trowel, or spoon)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Ziploc® bags
- Camera
- Tape measure
- Field logbook, surface soil field collection form, and pens
- Project-specific field sampling plan (FSP) and health and safety plan (HSP)
- Personal protective equipment (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Decontamination equipment.

PROCEDURES

1. Locate the sample station as directed in the project-specific FSP. Label containers with sample tags prior to filling in accordance with Integral's SOP on sample labeling (SOP-AP04). If analytical testing will be performed for volatile organic compounds (VOCs), collect the VOC sample first (with a minimum of disturbance) by placing the sample into the container with a minimum amount of headspace and sealed tightly.
2. Don a new pair of nitrile gloves and expose the soil surface by clearing an approximately 1 ft^2 area at the sampling site of any rocks or organic material greater than approximately 3 in. in size. Note any material removed from the sampling site in the field logbook.
3. Using a decontaminated stainless-steel sampling tool, excavate soil to the depth specified in the work plan.
4. If required for analysis, first collect VOC samples (prior to any homogenization) from a discrete location, placing the samples in the appropriate containers. Label sample containers before filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
5. Place additional sample material in a decontaminated plastic or stainless-steel mixing bowl.
6. Describe the soil in accordance with ASTM D2488-00 (see Integral's SOP on field classification of soils, SOP SL-04).
7. Thoroughly mix and homogenize the sample using disposable equipment or a decontaminated stainless-steel spoon until the color and texture are consistent throughout.
8. If required for analysis, first collect samples for grain-size tests before any large rocks are removed from the homogenized soil.
9. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized soil volume, note it on the surface soil field collection form or in the field logbook, and then discard the rocks.
10. Remove samples of the homogenized soil from the mixing bowl with the decontaminated stainless steel spoon and place in the appropriate size sample container. Do not touch the sample with your gloves. Fill the sample container with soil to just below the container lip, and seal the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling.
11. Mark the sampling site with a wire flag, wooden stake, metal rebar, or flagging, as appropriate.

12. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
13. Photograph sample location and document it in the logbook.
14. Decontaminate all sampling equipment according to Integral's SOP on decontaminating equipment for soil sampling (SOP SL-01) and in accordance with the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SL-06

LOGGING OF SOIL BOREHOLES

SCOPE AND APPLICATION

This SOP describes how to complete a Soil Boring Log form, which must be completed for Integral projects where soil boring techniques are performed during field exploration. A correctly completed form contains all of the information that must be recorded in the field to adequately characterize soil boreholes.

These procedures are adapted from ASTM D-2488-00. Field staff are encouraged to examine ASTM D-2488-00 in its entirety. This SOP represents minor modifications to emphasize environmental investigations rather than geotechnical investigations, for which the standards were written. Because each environmental project is unique and because job requirements can vary widely, the minimum standards presented may need to be supplemented with additional technical descriptions or field test results. However, all soil boring field logs, regardless of special project circumstances, must include information addressed in this SOP to achieve the minimum acceptable standards required by Integral.

LOG FORM INFORMATION

Project Number—Use the standard contract number.

Client—Identify the name of the client and the project site location.

Location—If stations, coordinates, mileposts, or similar markers are applicable, use them to identify the location of the project. If this information is not available, identify the facility (e.g., 20 ft NE of Retort #1).

Drilling Method—Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, cable tool) and the name of the drill rig (e.g., Mobil B 61, CME 55).

Diameter—Provide the diameter of the borehole. If the borehole has variable diameters, provide the depth interval for each diameter.

Sampling Method—Identify the type of sampler(s) used (e.g., standard split spoon, Dames & Moore sampler, grab).

Drilling Contractor—Provide the name of the drilling contractor.

Integral Staff—Enter the name(s) of Integral staff members performing logging and sampling activities.

Water Level Information—Provide the date, time, depth to static water, and casing depth. Generally, water levels should be taken each day before resuming drilling and at the completion of drilling. If water is not encountered in the boring, this information should be recorded.

Boring Number—Provide the boring number. A numbering system should be developed prior to drilling that does not conflict with other site information, such as previous drilling or other sampling activities.

Sheet—Number the sheets consecutively for each boring and continue the consecutive depth numbering.

Drilling Start and Finish—Provide the drilling start and finish dates and times.

For consecutive sheets, provide (at a minimum) the job number, boring number, and sheet number.

TECHNICAL DATA

Sampler Type—Provide the sampler type (e.g., SS = split spoon, G = grab).

Depth of Casing—Enter the depth of the casing below ground surface immediately prior to sampling.

Driven/Recovery—Provide the length that the sampler was driven and the length of sample recovered in the sampler. This column would not apply to grab samples.

Sample Number/Sample Depth—Provide the sample number. The sample numbering scheme should be established prior to drilling. One method is to use the boring number and consecutive alphabetical letters. For instance, the first sample obtained from boring MW-4 would be identified as 4A, the second would be identified as 4B, and so on. Another method for sample identification is naming the boring number with the depth. For example, the sample from Boring 1 at 10 ft would be labeled B1-10'. The depth of the sample is the depth of the casing plus the length to the middle of the recovered sample to the nearest 0.1 ft. Typically, split spoon samplers are 18 in. long. Samples should be obtained from the middle of the recovered sample. The depth of the sample with the casing at 10 ft would then be 10.7 ft.

Number of Blows—For standard split-spoon samplers, record the number of blows for each 6 in. of sampler penetration. A typical blow count of 6, 12, and 14 is recorded as 6/12/14. Refusal is a penetration of less than 6 in. with a blow count of 50. A partial penetration of 50 blows for 4 in. is recorded as 50/4". Total blows will be recorded for nonstandard split spoons (e.g., 5-ft tube used for continuous sampling).

Blank Columns—Two blank columns are provided. Use these columns for site-specific information, usually related to the chemicals of concern. Examples for a hydrocarbon site would be sheen and photoionization detector readings of the samples.

Depth—Use a depth scale that is appropriate for the complexity of the subsurface conditions. The boxes located to the right of the scale should be used to graphically indicate sample locations as shown in the example.

Surface Conditions—Describe the surface conditions (e.g., paved, 4-in. concrete slab, grass, natural vegetation and surface soil, oil-stained gravel).

Soil Description—Enter the soil classification and definition of soil contacts using the format described in SOP SL-04, *Field Classification of Soil*.

Comments—Include all pertinent observations. Drilling observations might include drilling chatter, rod-bounce (boulder), sudden differences in drilling speed, damaged samplers, and malfunctioning equipment. Information provided by the driller should be attributed to the driller. Information on possible contaminants might include odor, staining, color, and presence or absence of some indicator of contamination. Describe what it is that indicates contamination (e.g., fuel-like odor, oily sheen in drill cuttings, yellow water in drill cuttings).

ATTACHMENT 1. SOIL BORING LOG FORM

integral consulting inc.
319 SW Washington St., Suite 1150
Portland, OR 97204
(503) 284-5545

STATION NUMBER
PROJECT
LOCATION
PROJECT NUMBER
LOGGED BY

SAMPLE INFORMATION						STRATA	DESCRIPTION
Sample ID	Depth	Time	Tag No.	% Recov.	Depth (Feet)		USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, weathering, cementation, geologic interpretation, etc.
					2--		
					4--		
					6--		
					8--		
					10--		
					12--		
					14--		

DRILLING CONTRACTOR
DRILLING METHOD
SAMPLING EQUIPMENT
DRILLING STARTED
COORDINATES
SURFACE ELEVATION
DATUM

Location Sketch

ATTACHMENT 2. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-µm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved Feb. 10, 2000. Published May 2000. Originally published as D 2488 – 66 T. Last previous edition D 2488 – 93$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols.*

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula*.

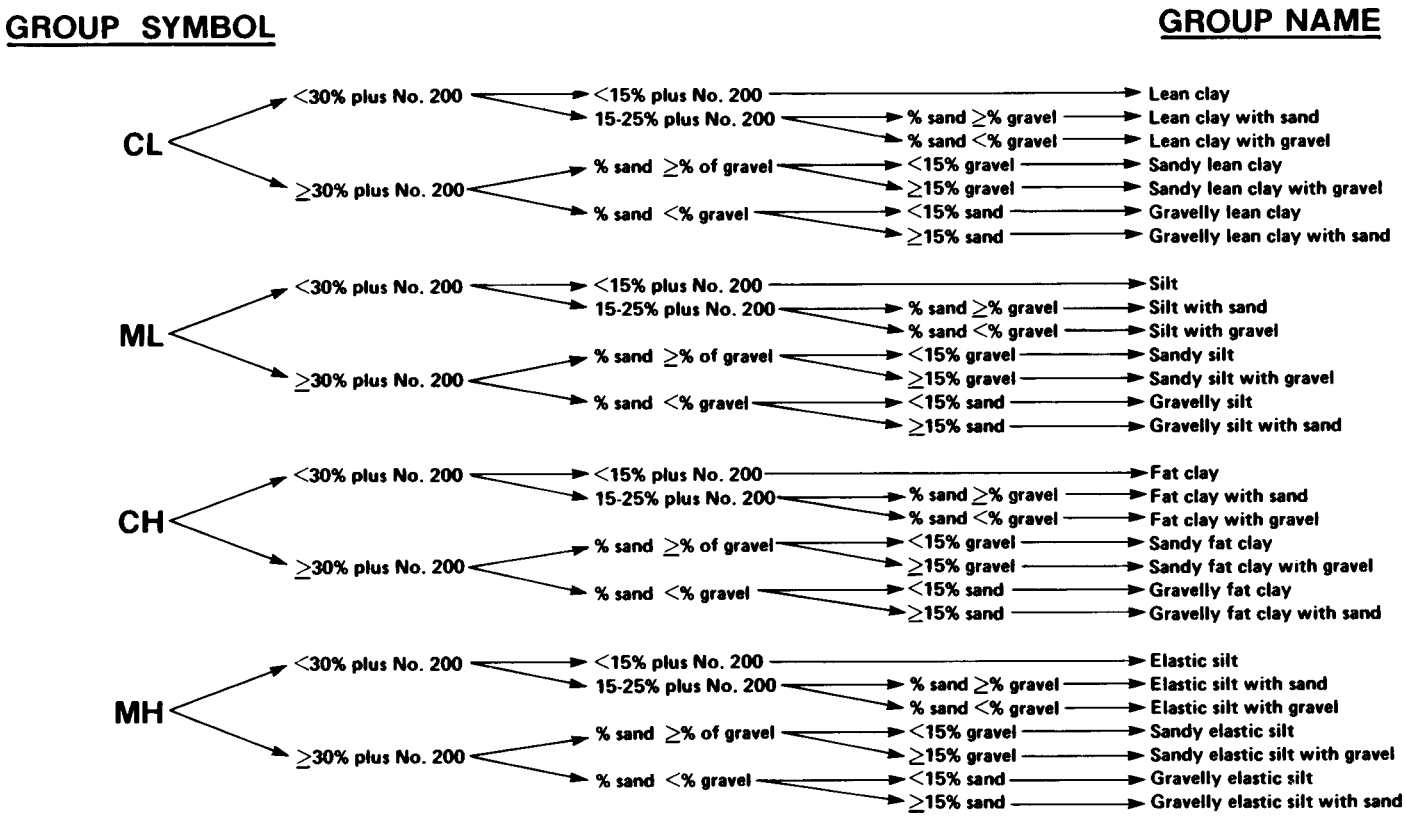
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens*.

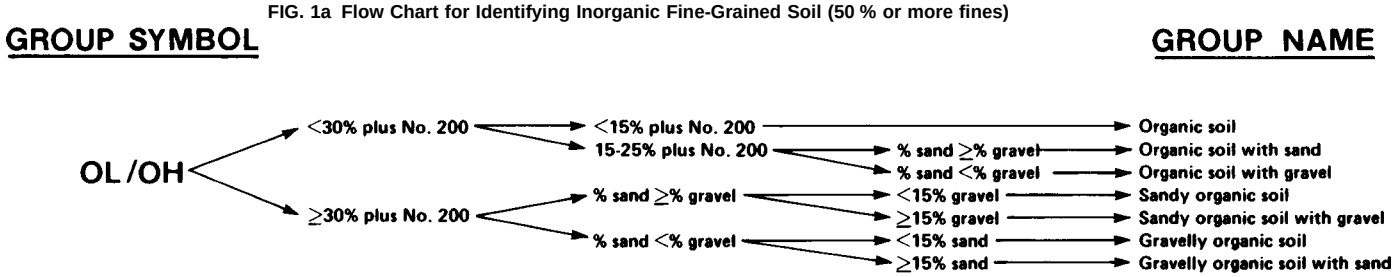
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

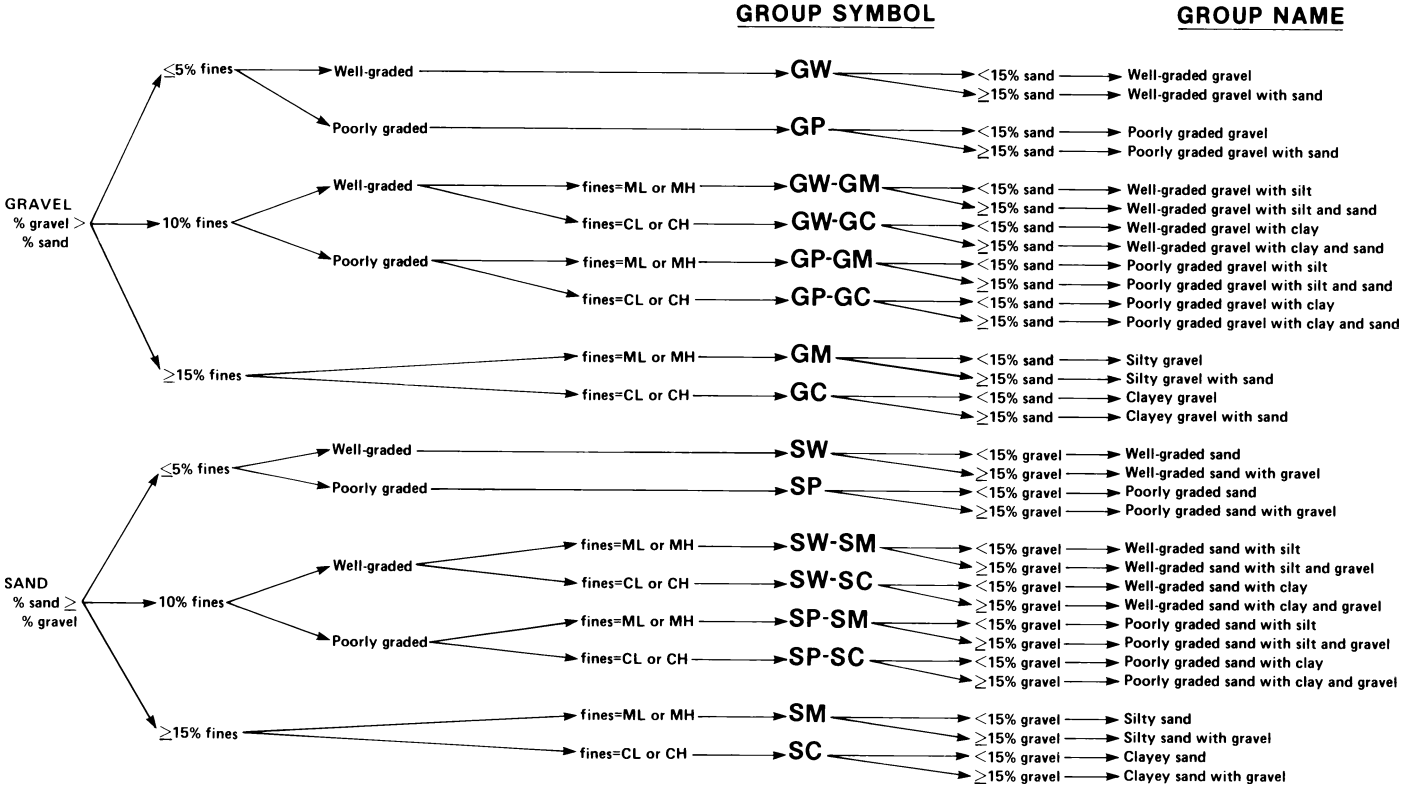
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

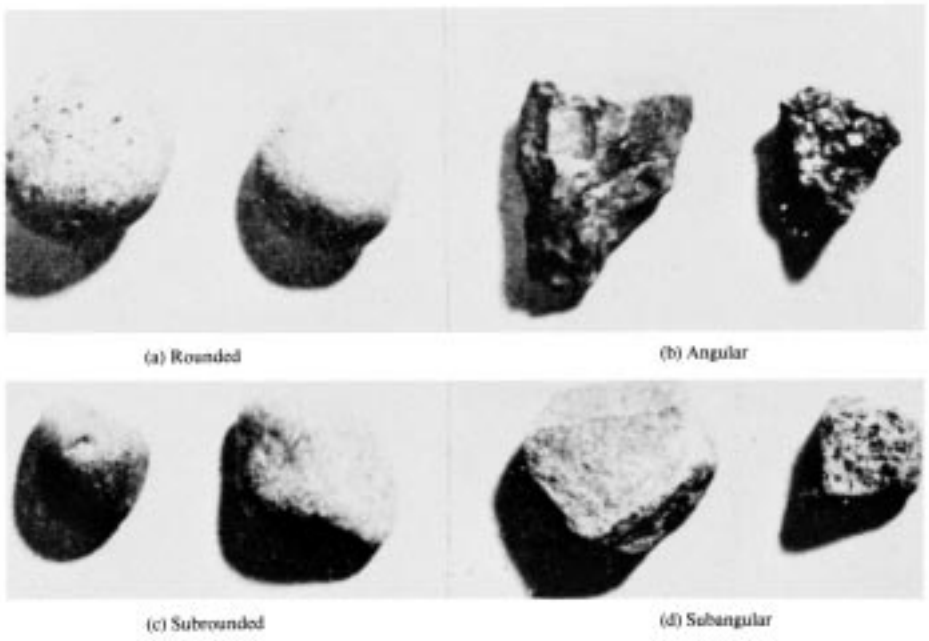


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

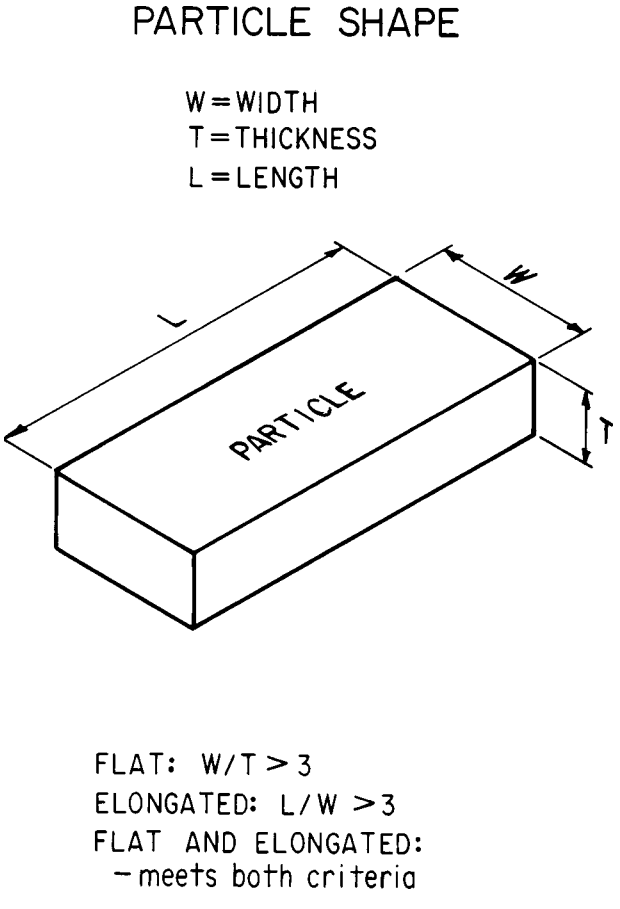


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as “Sandy Lean Clay (CL)”; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; “Poorly Graded Sand with Silt (SP-SM)”; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken

shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993$^{\epsilon 1}$) that may impact the use of this standard.

(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

ATTACHMENT A3

FIELD FORMS

LIST OF FIELD FORMS

Soil Borehole Log

Chain of Custody/Laboratory Analysis Request Form

Field Change Request and Corrective Action Record

integral consulting inc.
411 1st Avenue South, Suite 550
Seattle, WA 98104
(206) 230-9600

BORING NUMBER _____
PROJECT _____
LOCATION _____
PROJECT NUMBER _____
LOGGED BY _____

SAMPLE INFORMATION						STRATA	DESCRIPTION
Sample Identifier	Sample Number	Time	Percent Recovery		Depth (Feet)		USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, weathering, cementation, geologic interpretation, etc.
					1--		
					2--		

ADDITIONAL NOTES / SKETCHES

FIELD PERSONNEL _____
SAMPLING METHOD _____
SAMPLING EQUIPMENT _____
DATE _____
TIME STARTED _____
TIME ENDED _____
COORDINATES _____

Location Sketch

CHAIN OF CUSTODY/LABORATORY ANALYSIS REQUEST FORM

Page ______ of ______ Turn Around Requested: ________

Report to:	Phone:				Analyses Requested										Notes/Comments
Company:	Fax:														
Address:	Proj Name:														
City:	Proj Number:														
State:	Sampler:														
Zipcode:															
Sample ID	Sample Date	Sample Time	Sample Matrix	No. Containers											

Relinquished: (Signature)	Received by: (Signature)	Special Instructions/Notes	
Printed name:	Printed name:		
Company:	Company:		Number of Coolers:
			Cooler Temp(s):
Date: Time:	Date: Time:		COC Seals Intact?
			Bottles Intact?

	FIELD CHANGE REQUEST	Project Number:

Field Change No.
Page _____ to

Project Number:
Project Name:

CHANGE REQUEST
Applicable Reference:
Description of Change:

Reason for Change:

Impact on Present and Completed Work:

Requested by:
Date: ____/____/

(Field Scientist)

Acknowledged by:
Date: ____/____/

(Field Task Leader)

FIELD OPERATIONS MANAGER RECOMMENDATION

Recommended Disposition:

Recommendation by:
Date: ____/____/

(Sampling and Analysis Coordinator)

PROJECT MANAGER APPROVAL

Final Deposition:

Approved/Disapproved by:
Date: ____/____/

(Project Coordinator)

CORRECTIVE ACTION RECORD

Page ___ of

Audit Report No. :_____ Date:

Report Originator:

Person Responsible for Response:

DESCRIPTION OF PROBLEM:

Date and Time Problem Recognized: _____ By:

Date of Actual Occurrence: _____ By:

Analyte: _____ Analytical Method:

Cause of Problem:

CORRECTIVE ACTION PLANNED:

Person Responsible for Corrective Action:

Date of Corrective Action:

Corrective Action Plan Approval: _____ Date:

DESCRIPTION OF FOLLOW-UP ACTIVITIES:

Person Responsible for Follow-up Activities:

Date of Follow-up Activity:

Final Corrective Action Approval: _____ Date:

ATTACHMENT A4

USEPA RISK ASSESSMENT GUIDANCE FORMS (PER THE UNILATERAL ADMINISTRATIVE ORDER STATEMENT OF WORK)

EXHIBIT 5

USEPA SAMPLING DESIGN SELECTION WORKSHEETS

Exhibit 5. Part I: Medium Sampling Summary
Sampling Design Selection Worksheet

A. Site Name San Jacinto River Waste Pits **B. Base Map Code**

C. Medium: Groundwater, Soil, Sediment, Surface Water, Air or Other (specify) Soil

D. Comments Soil samples associated with the monitoring well borings are not included on this form, because the number of samples is currently unknown. These well-boring soils will be analyzed for grain size, and archived for chemistry.

E. Medium/ Pathway Code	Exposure Pathway/ Exposure Area Name	F. Number of Samples from Part II					
		Judgmental/ Purposive	Background	Statistical Design	Geometrical or Geostatistical Design	QC	Row Total
Soil	Nature and extent, exposure assessments, contaminant fate and transport	62	40	NA	NA	14	116
	Column Totals:	62	40	NA	NA	14	116
					G. Grand Total:		116

Exhibit 5. Part II: Exposure Pathway Summary

Sampling Design Selection Worksheet (cont'd.)

H. Radionuclide of Potential Concern and CAS Number	I. Frequency of Occurrence	J. Estimation		K. CV	L. Background
		Arithmetic Mean	Maximum		
NA	NA	NA	NA	NA	NA

M. Code (CAS Number) of Radionuclide of Potential Concern Selected as Proxy NA

N. Reason for Defining New Stratum or Domain (check one)

- ☐ Heterogeneous Radionuclide Distribution
- ☐ Geological Stratum Controls
- ☐ Historical Information Indicates Difference
- ☐ Field Screening Indicates Difference
- ☐ Exposure Variations
- ☒ Other (specify) NA

Q. Stratum or Exposure Area	P. Reason	Q. Number of Samples from Part III					
Name and Code		Judgmental/ Purposive	Background	Statistical Design	Geometrical or Geostatistical Design	QC	Row Total
Soil	Nature and extent, exposure assessments, contaminant fate and transport	62	40	NA	NA	14	116
R. Total (Part I, Step F):			40	NA	NA	14	116

Exhibit 5. Part III: Exposure Area Summary
Sampling Design Selection Worksheet (cont'd.)

O. Stratum or Exposure Area San Jacinto River Waste Pits **Domain Code**

E. Medium/Pathway Code Soil **Pathway Code**

S. Judgmental or Purposive Sampling

Comments

Use prior site information to place samples, or determine location and extent of contamination. Judgmental or purposive samples generally cannot be used to replace statistically located samples. An exposure area and stratum MUST be sampled by at least TWO samples.

Number of Samples 116

T. Background Samples

Background samples must be taken for each medium relevant to each stratum/area. Zero background samples are not acceptable. See the discussion on pp. 74-75 of Guidance for Data Useability in Risk Assessment Part A.

Number of Background Samples 20

U. Statistical Samples

CV of proxy or radionuclide of potential concern NA

Minimum Detectable Relative Difference (MDRD) NA (<40% if no other information exists)

Confidence Level NA (>80%) Power of Test NA (>90%)

Number of Samples (See formula in Appendix IV) NA

V. Geometrical Samples

Hot spot radius NA Enter distance units) NA

Probability of hot spot prior to investigation NA (0 to 100%)

Probability that NO hot spot exists after investigation (See formula in Appendix IV) NA (enter only if >75%)

W. Geostatistical Samples

Required number of samples to complete grid + number of short range samples NA

X. Quality Control samples

Number of Duplicates (Minimum 1:20 environmental samples) 5

Number of Blanks (Minimum 1 per medium per day or 1 per sampling process, whichever is greater) 9

Y. Sample Total for Stratum (Part II, Step U)

Judgmental/ Purposive	Background	Statistical Design	Geometrical or Geostatistical Design	QC	Row Total
62	40	NA	NA	14	116

EXHIBIT 52

METHOD SELECTION WORKSHEET

Exhibit 52. Method Selection Worksheet

I. Analytes		II. Medium	III. Critical parameters				IV. Routine Available Methods[4]
A. Chemical or Class of Chemicals of Potential Concern	B. Reporting Requirement[1] (Y/N)		A. Turnaround Time (enter hours or days)	B. ID Only or ID Plus Quant (ID or ID+Q)	C. Concentration of Concern (or PRG)[2]	D. Required Method Detection Limit[3]	
Dioxins/furans	N	Soil	21 days	ID+Q	2,3,7,8-TCDD TEQ of 17 ng/kg	Not applicable	1613B
Aluminum	N	Soil	21 days	ID+Q	990,000 mg/kg	198,000 mg/kg	6010B / 6020
Antimony	N	Soil	21 days	ID+Q	410 mg/kg	82 mg/kg	6010B / 6020
Arsenic	N	Soil	21 days	ID+Q	1.6 mg/kg	0.32 mg/kg	6010B / 6020
Barium	N	Soil	21 days	ID+Q	190,000 mg/kg	38,000 mg/kg	6010B / 6020
Cadmium	N	Soil	21 days	ID+Q	800 mg/kg	160 mg/kg	6010B / 6020
Chromium	N	Soil	21 days	ID+Q	1,500,000 mg/kg	300,000 mg/kg	6010B / 6020
Cobalt	N	Soil	21 days	ID+Q	300 mg/kg	60 mg/kg	6010B / 6020
Copper	N	Soil	21 days	ID+Q	41,000 mg/kg	8,200 mg/kg	6010B / 6020
Lead	N	Soil	21 days	ID+Q	800 mg/kg	160 mg/kg	6010B / 6020
Magnesium	N	Soil	21 days	ID+Q	No value	Not applicable	6010B / 6020
Manganese	N	Soil	21 days	ID+Q	23,000 mg/kg	4,600 mg/kg	6010B / 6020
Nickel	N	Soil	21 days	ID+Q	20,000 mg/kg	4,000 mg/kg	6010B / 6020
Silver	N	Soil	21 days	ID+Q	5,100mg/kg	1020 mg/kg	6010B / 6020
Thallium	N	Soil	21 days	ID+Q	78 mg/kg	15.6 mg/kg	6010B / 6020
Vanadium	N	Soil	21 days	ID+Q	72 mg/kg	14.4 mg/kg	6010B / 6020
Zinc	N	Soil	21 days	ID+Q	310,000 mg/kg	62,000 mg/kg	6010B / 6020
Mercury	N	Soil	21 days	ID+Q	34 mg/kg	6.8 mg/kg	7471A
PCB 77	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A

I. Analytes		II. Medium	III. Critical parameters				IV. Routine Available Methods[4]
A. Chemical or Class of Chemicals of Potential Concern	B. Reporting Requirement[1] (Y/N)		A. Turnaround Time (enter hours or days)	B. ID Only or ID Plus Quant (ID or ID+Q)	C. Concentration of Concern (or PRG)[2]	D. Required Method Detection Limit[3]	
PCB 81	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
PCB 105	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
PCB 114	N	Soil	21 days	ID+Q	2.3 µg/kg	0.46 µg/kg	1668A
PCB 118	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
PCB 123	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
PCB 126	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
PCB 156	N	Soil	21 days	ID+Q	230 µg/kg	46 µg/kg	1668A
PCB 157	N	Soil	21 days	ID+Q	230 µg/kg	46 µg/kg	1668A
PCB 167	N	Soil	21 days	ID+Q	1,100 µg/kg	220 µg/kg	1668A
PCB 169	N	Soil	21 days	ID+Q	1.1 µg/kg	0.22 µg/kg	1668A
PCB 189	N	Soil	21 days	ID+Q	110 µg/kg	22 µg/kg	1668A
Total PCBs	N	Soil	21 days	ID+Q	No value	Not applicable	1668A
Acenaphthene	N	Soil	21 days	ID+Q	33,000,000 µg/kg	6,600,000µg/kg	8270C
Fluorene	N	Soil	21 days	ID+Q	22,000,000 µg/kg	4,400,000µg/kg	8270C
Naphthalene	N	Soil	21 days	ID+Q	18,000 µg/kg	3,600 µg/kg	8270C
Phenanthrene	N	Soil	21 days	ID+Q	19,000,000 µg/kg	3,800,000 µg/kg	8270C
2,4,6-Trichlorophenol	N	Soil	21 days	ID+Q	160,000 µg/kg	32,000 µg/kg	8270C
2,4-Dichlorophenol	N	Soil	21 days	ID+Q	180,000 µg/kg	36,000 µg/kg	8270C
Pentachlorophenol	N	Soil	21 days	ID+Q	9,000 µg/kg	1,800 µg/kg	8270C
Phenol	N	Soil	21 days	ID+Q	180,000,000 µg/kg	36,000,000 µg/kg	8270C
Hexachlorobenzene	N	Soil	21 days	ID+Q	1,100 µg/kg	220 µg/kg	8270C
2,3,4,6-Tetrachlorophenol	N	Soil	21 days	ID+Q	18,000,000 µg/kg	3,600,000 µg/kg	8270C

I. Analytes		II. Medium	III. Critical parameters				IV. Routine Available Methods[4]
A. Chemical or Class of Chemicals of Potential Concern	B. Reporting Requirement[1] (Y/N)		A. Turnaround Time (enter hours or days)	B. ID Only or ID Plus Quant (ID or ID+Q)	C. Concentration of Concern (or PRG)[2]	D. Required Method Detection Limit[3]	
Carbazole	N	Soil	21 days	ID+Q	950,000 µg/kg	190,000 µg/kg	8270C
2,4,5-Trichlorophenol	N	Soil	21 days	ID+Q	62,000,000 µg/kg	12,400,000 µg/kg	8270C
Bis(2-ethylhexyl) phthalate	N	Soil	21 days	ID+Q	120,000 µg/kg	24,000 µg/kg	8270C
Chloroform	N	Soil	21 days	ID+Q	1,500 µg/kg	300 µg/kg	8260B
1,2,4-Trichlorobenzene	N	Soil	21 days	ID+Q	270,000 µg/kg	54,000 µg/kg	8260B
1,2-Dichlorobenzene	N	Soil	21 days	ID+Q	9,800,000 µg/kg	1,960,000 µg/kg	8260B
1,3-Dichlorobenzene	N	Soil	21 days	ID+Q	88,000 µg/kg	17,600 µg/kg	8260B
1,4-Dichlorobenzene	N	Soil	21 days	ID+Q	12,000 µg/kg	2,400 µg/kg	8260B
1,2,3-Trichlorobenzene	N	Soil	21 days	ID+Q	490,000 µg/kg	98,000 µg/kg	8260B

[1]Y = total reported for compound class
N = each analyte reported separately
[2]Preliminary remediation goal
[3]Method detection limit should b no greater than 20% of concentration of concern
[4]Refer to Appendix III for specific methods. Recommend consultation with chemist and/or automated methods search to determine all methods available. (Exhibit 53 lists computer systems that support method selection.

APPENDIX B
LABORATORY QUALITY ASSURANCE MANUAL AND STANDARD OPERATING PROCEDURES

TARGET SHEET

SITE NAME: SAN JACINTO RIVER WASTE PITS

CERCLIS I.D.: TXN000606611

TITLE OF DOC.: DRAFT SAMPLING AND ANYLSIS PLAN: SOIL STUDY

DATE OF DOC.: 12/01/2010

NO. OF PGS. THIS TARGET SHEET REPLACES: 81

SDMS #: 9546106 **RELATED #:** 9184235

SENSITIVE ?	X	**MISSING PAGES ?**	
ALTERN. MEDIA ?		**CROSS REFERENCE ?**	

LAB DOCUMENT ?		**LAB NAME:**	
ASC./BOX #:			
CASE #:		**SDG #:**	

COMMENTS : PAGES 289-369 WERE REDACTED FROM THIS DOCUMENT DUE TO FOIA EXEMPTION 4 - CONFIDENTIAL BUSINESS INFORMATION

STANDARD OPERATING PROCEDURE

for

DATA ARCHIVING

SOP No.: ADM-ARCH

Revision 4

August 7, 2008

Approved by: _____ 8/8/08
Client Services Manager — Date

_____ 8-8-08
QA Manager — Date

_____ 8/8/08
Laboratory Director — Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
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NUMBER: _____
Initials: _____ Date: _____

DATA ARCHIVING

1 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes a standard system for archiving laboratory data and associated records for Columbia Analytical Services' Kelso, Wa. facility. This procedure applies to laboratory data kept in service request (project) files and to data organized and bound by laboratory sections. The procedure also describes a system for retrieving archived data for later review or submittal to clients.

1.2 Backup and archiving of electronic data is not addressed in this procedure. Refer to the CAS Software Quality Assurance Plan and the *SOP for Tape Backup and Tape Archiving*.

1.3 This SOP does not apply to EPA Contract Laboratory Program data archiving, which has distinct data archiving procedures defined in the Statement of Work for the contract. For those procedures, refer to the CLP category of SOPs.

2 SUMMARY OF SYSTEM

2.1 A defined system for archiving data is required in order to provide a permanent record of all analytical procedures used in a given sample analysis. Also, it is important to maintain a permanent record of the analytical report. Archived records must provide all information needed to reconstruct the entire laboratory process.

2.2 It is laboratory policy to archive all sample handling records, analytical reports, raw data, and associated records applicable to all analyses performed. Certain records, such as project supporting documentation and laboratory logbooks, will be retained in hard copy form. Analytical reports may be retained in either hard copy or electronic (pdf) form. Laboratory records (typically raw data) associated with multiple service requests or projects is bound by the laboratory staff and submitted to the records staff. Service request files are routed to the records clerk as a function of the post-reporting system. The service request files will contain all project supporting documentation and (when hard copy) a copy of the analytical report and the sample raw data. Service request files are then archived in a sequential file system by service request number. This includes a set of files for short-term holding/staging and permanent long term storage.

2.3 The default data retention period to archived data is five (5) years from the completion of a project. However, binding contracts or project requirements, programs, or regulations may specify a shorter or longer period. For example, for projects originating in Alaska or Louisiana, the record retention time is 10 years. For those contracts with longer retention periods than the 5 year period, clients will be given the opportunity to take possession of their documents, or CAS will retain them for the required period.

2.4 Once the retention period has elapsed, documents associated with the "expired" projects will be destroyed. The lab will dispose of the data unless the client makes arrangements to take possession of it.

3 RESPONSIBILITIES

3.1 It is the responsibility of the laboratory staff submitting reports and supporting data to a service request file to provide all data directly related to an analysis. This is essential so that the analysis can be reconstructed from the archived data. When certain data pertains to multiple analyses (calibration, tuning, blank data, etc.) the lab must have systems in place to provide copies of this data or archive this data separately.

3.2 Throughout the process of creating, assembling, copying, and mailing the analytical report, all staff involved (filing, report production, project chemist, copy center) are responsible for the service request file contents as a whole. Steps must be taken to ensure that all data required in the file is kept in the file.

3.3 It is the responsibility of the records staff to maintain the archiving system and to follow this procedure. Staff members must be properly trained on its use.

3.4 It is the responsibility of project chemists to inform the records staff of data retention requirements (timeframes, notifications, etc.) that differ from standard procedures.

4 DEFINITIONS

Analytical report - The Certified Analytical Report generated by the laboratory, in final form as delivered to the client, in both hard copy and electronic formats.

LIMS - CAS Laboratory Information Management System

Raw data - Any hard copy laboratory data associated with a given analysis. May be archived with the service request file or separately.

Service Request File (SR) - This is the physical file which contains all documentation associated with a given set of samples (chain of custody, sample receipt information, etc.). The service request identifies the analyses and reporting to be done for the sample(s). The CAS LIMS assigns a unique number to each service request. Also known as report file or project file.

5 PROCEDURE

5.1 Data Retention Period

5.1.1 The standard data retention time for analytical reports, SR files and laboratory data records is 5 years unless otherwise noted in Tables 1 and 2. Changes to Tables 1 and 2 can be made on the SOP Change Form accompanying the distributed SOP, and are to be incorporated into future revisions of this SOP.

NOTE: Longer client or project record retention may supersede agency requirements.

5.1.2 Table 1 lists data retention rules for different state and federal programs. Records falling under these states or programs must be held for the time period listed before disposal.

5.1.3 Table 2 lists data retention rules for various clients and projects. Records for these must be held for the time period listed before disposal.

5.1.4 Quality Assurance records (section 5.4) are held indefinitely.

5.2 Service Request Files

5.2.1 After the copy center has scanned and/or copied and shipped the analytical report, the SR file is forwarded to the clerk performing hazardous materials review. The LIMS provides a record of report shipment and completion

5.2.2 Following shipment and hazardous materials review, the SR file is forwarded to the records clerk. Out cards are provided for any files that may have been checked out during the process.

5.2.3 The content of the data file is verified to ensure that all necessary records are included. The following contents (in general order) are accounted for:

- File tracking sheet(s)
- EDD sheet
- Hazardous review sheet
- Chain of custody (if not included in report)
- Report pages (when archived hard copy)
- Lab generated data (raw data), general chemistry followed by metals followed by organics.
- Miscellaneous sheets/documents (instructions, notes, phone logs, etc.)
- SR acknowledgment
- Internal COC.

5.2.4 Analytical reports archived electronically are kept in the R:\copy network directory. A tape backup is performed each day, and tapes are handled as described in the SOP for Electronic Data Backup and Archiving. As the directory fills, the electronic reports are moved to DVD and stored in a designated area.

5.2.5 Short term holding/staging

5.2.5.1 Report files are centrally located in the records room file cabinet bank. Large files (approximately 500 pages or more) are placed on shelves near the file bank.

5.2.5.2 To check out a file from this area, the file is replaced with an out card indicating the date, service request number, and name or person it is checked out to. If the file has been checked out prior to arriving in the records area, an out card is placed in the file hanger (in portable file) where file would be located.

5.2.5.3 Short term report holding is typically for a period of 4-6 weeks, depending on the number and size of files.

5.2.6 Long term archiving

5.2.6.1 After the period of short term holdng (when the oldest dated file drawer in short term holding is full) the files are prepared for long term archiving. The SR files are archived in separate, secured off-site facilities within close proximity of the laboratory.

5.2.6.2 Organize the files in cardboard file boxes, within original manila file folders or expandable file folders.

5.2.6.3 The SR files are placed in file boxes in approximate numerical order and the file boxes are numbered sequentially. A File Archive Record listing the box number, files in the box, and file size (for larger files) is maintained in a 3-ring binder Exceptions to the numerical order are necessary due to large file sizes. These exceptions are listed on the archive record sheet.

5.2.6.3.1If an out card is used, indicating that a file has been checked out, record this in the 3-ring binder. An out card is placed in the box in place of the file. The information recorded in the 3-ring binder includes when the file was checked out, client, who checked the file out, service request number, and project chemist's name. When the SR file is returned, the out card is removed.

5.2.6.3.2Write the year on the end of the file box along with the sequence of file numbers contained in file box. Boxes of files are transferred to the long-term storage facility All boxes are stored in numerical order.

5.2.6.4 The long term archiving storage is a controlled-access area. Records are protected from fire, theft, loss, environmental deterioration, and vermin. The area is locked at all times when not in use. An electronic access log is maintained for long term archiving storage. This is located on R:\Copy\Fileroom\Archived Files Access Log. Each time access is made to a storage area, the person accessing will complete the information on the form.

5.2.6.5 For retrieval of SR files, the checkout system is the same out card system as used with short term storage, with an out card left in the file box.

5.3 Laboratory Data Records

5.3.1 Laboratory records (typically raw data) associated with multiple service requests or projects is bound by the laboratory staff and submitted to the records clerk. These data books are labeled according to instrument location and/or lab section.

5.3.2 Lab instrumentation books are transferred to the storage/archiving area. They are stored in file boxes, the boxes labeled and placed on labeled shelves. A record is kept to catalog the data books. The clerk maintains a hardcopy record and an electronic copy is kept.

5.3.3 The long term archiving storage is a controlled-access area off-site. Records are protected from fire, theft, loss, environmental deterioration, and vermin. The area is locked at all times when not in use.

5.3.4 The check out system for the lab data books is the same as for file check out. The out card system is used by the records clerk. The out card will indicate who checked out the book, when the book was checked out and exactly which book was checked out. The out card is placed in the box or on the shelf in place of the book.

5.4 Quality Assurance Records

5.4.1 The QA staff periodically performs archiving of lab-wide quality assurance records. This includes certification records, controlled documents (historical SOPs, logbooks, etc.), training records, PT testing results, audit records, QAPPs, metrology records, etc. The QA staff places the records in sequentially numbered file boxes. An electronic record of the file box contents is maintained.

5.4.2 Archived QA records are stored in the long term data archiving storage units. Records are protected from fire, theft, loss, environmental deterioration, and vermin. The area is locked at all times when not in use.

5.5 Record Disposal

5.5.1 Following the data retention period (see section 5.1) data may be destroyed. In some cases, the client will require notification prior to disposal. In this case, document any cases where the records are to be held longer or provided to the client.

5.5.2 Records are disposed of by emptying file boxes and file folders into a dumpster. The files boxes, flex folders, file folders, etc are recycled for reuse.

5.6 Plan for Ownership Change or Laboratory Closure

5.6.1 In the event that laboratory ownership changes or the laboratory closes, it is expected that the disposition of laboratory records will be addressed in the legal documents associated with the change. The laboratory will notify clients of the change and will follow any instructions obtained from clients regarding archived records. This may include keeping the records in storage (transferring to the new ownership), transferring to the client, destroying them, or other options. Additionally, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

5.6.2 If there is a change in ownership all records and analyses performed pertaining to NELAP and DoD accreditation/approval will be kept for a minimum of 5 years and will be made available for inspection by the accrediting authorities during this period.

6 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

A review of the data archiving system will be included as part of internal auditing of data and reports conducted by quality assurance staff. Adherence to the procedures described in this SOP will be assessed. Any deficiencies uncovered will require corrective action as described in applicable audit reports.

7 RECORDS

Documentation and recordkeeping requirements are listed in Section 5.

8 REFERENCES

8.1 Quality Assurance Manual, Revision 17, Columbia Analytical Services, Inc., January 30, 2008

8.2 National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.

8.3 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

9 CHANGES FROM PREVIOUS REVISION

9.1 Section 2.2 revised.

9.2 New Section 5.2.4.

9.3 Section 5.2.5.4, added access log requirement and details.

9.4 Section 5.4.2, corrected QA storage area from upstairs corporate to off-site.

9.5 New Section 5.6

9.6 Section 6 revised to add current QA Manual, NELAP, and QSM references.

9.7 Minor formatting and typographical corrections.

TABLE 1
Agency Record Retention Requirements

Federal/National Programs	Record Retention (yrs)	Comments
NFESC/Navy	10	Notify prior to disposal
U.S. Air Force (AFCEE)	5	
U.S. Army Corps of Engineers	5	
State Programs		
Alaska UST	10	
Arizona	5	DW*
Arkansas	(standard)	Record retention not found in certification rules
California	(standard)	Record retention not found in certification rules DW*
Colorado	5	DW*
Florida	5	DW*
Hawaii	10	DW*
Idaho	5	DW*
Indiana	5	DW*
Louisiana	10	DW*
Maine	(standard)	Record retention not found in certification rules DW*
Michigan	(standard)	Record retention not found in certification rules DW*
Minnesota	3 / 10	3 yrs for wastewater, 10 yrs for drinking water
Montana	5	DW*
Nevada	5	DW*
New Jersey	5 / 10	10yrs if lab is informed analyses are due to public health concerns
New Mexico	5	DW*
New York	6	
North Carolina	5	
Oklahoma	3 / 10	10 yrs for municipal water testing
Oregon	5	DW*
South Carolina	(standard)	Record retention not found in certification rules
Utah	5	DW*
Washington	(standard)	Record retention not found in certification rules DW*
Wisconsin	3	DW*
Wyoming (EPA Region8)	5	DW*

* The EPA *Manual for the Certification of Laboratories Analyzing Drinking Water* states that "Public Water Systems are required to maintain records of chemical analyses of compliance samples for 10 years (40 CFR 141.33) and lead and copper for 12 years (40 CFR 141.91). The laboratory should maintain easily accessible records for five years or until the next certification data audit is complete, whichever is longer. The client water system should be notified before disposing of records so they may request copies if needed."

TABLE 2
Client/Project Record Retention Requirements

COMPNAME	Data Retention Years	Data Retention Notify
Camp Dresser & McKee, Incorporated (CDM)	5	Y
Hart Crowser, Incorporated	6	
Portland, City of	5	Y
Beazer East, Inc.	7	Y
URS Corporation	5	Y
Landau Associates,Inc.	6	
GeoTrans, Inc.	7	
ARCADIS of New York, Inc.	5	Y
Exponent	5	Y
Barr Engineering Company	5	Y
Bechtel Environmental, Incorporated	7	
Portland, Port of	5	Y
Brown and Caldwell	0	
Menzie-Cura Associates	5	Y
Cosmopolitan Engineering Group	5	Y
Tetra Tech EM, Incorporated	5	Y
Science Application International Corporation (SAIC)	5	Y
US Army Corps Of Engineers	5	Y
Metcalf & Eddy, Incorporated	10	
Environmental Quality Management	10	
Jacobs Engineering Group, Incorporated	10	Y
Integral Consulting, Incorporated	5	Y
Normandeau Associates	6	
SECOR International, Incorporated	7	
Malcolm Pirnie, Incorporated	10	
Environet, Incorporated	10	
Shaw Environmental & Infrastructure, Inc.	7	Y
TEC, Inc. - The Environmental Company,Inc.	10	
Montgomery Watson Americas, Incorporated	10	

STANDARD OPERATING PROCEDURE

AUTOMATED SOXHLET EXTRACTION

EXT-3541

Revision 6

September 28, 2009

Approved By:

Supervisor		9/24/09 Date
QA Manager		9/25/09 Date
Laboratory Manager		9/25/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
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NUMBER: _____

Initials: _____ Date: _____

AUTOMATED SOXHLET EXTRACTION

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in EPA Method 3541 for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sediments, sludges, wastes, and tissues.

1.2. This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

2. SUMMARY OF METHOD

2.1. This procedure describes the automated Soxhlet extraction process. The Soxhlet extraction ensures intimate contact of the sample matrix with the extraction solvent over a period of time. The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble above a plug of glass wool, and extracted using an appropriate solvent on a Soxtherm extractor for a prescribed amount of time.

2.2. Following the extraction period, the resulting extract is then dried if necessary, concentrated, and as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3. INTERFERENCES

3.1. Phthalate esters can pose difficulties when performing sample extractions for organochlorine pesticides, PCBs, and other semi-volatile organics. Phthalates are easily extracted or leached from materials containing plastics during laboratory operations. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials.

3.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. All apparatus must be cleaned prior to use on individual samples.

3.3. Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most OP pesticides will degrade in this situation. All glassware must be rinsed very carefully to avoid this problem.

3.4. Refer to SW-846 Method 3500 for additional discussion of interferences. Additional cleanup procedures are described in the applicable CAS SOP.

4. SAFETY

4.1. All appropriate safety precautions for handling solvents, reagents, and samples must be taken when performing this procedure. This includes the use of protective equipment (safety glasses, lab coats, gloves, etc.) and use of correct glassware handling practices.

4.2. Chemicals, reagents, standards, and samples must be handled as described in CAS safety policies, approved methods, and in MSDSs where available. Refer to the specific analytical method and the CAS Safety Manual for guidance.

5. RESPONSIBILITIES

5.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

5.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the determinative method, is also the responsibility of the department supervisor/manager.

6. APPARATUS AND MATERIALS

6.1. Soxtherm automated extraction system

6.1.1. Extraction unit controller

6.1.2. Beakers, 54 x 130mm

6.1.3. Thimbles, 33mm inner diameter, glass or cellulose

6.1.4. Metal thimble holder

6.2. Chiller unit

6.3. Boiling chips – Teflon, pre-cleaned by rinsing or extraction.

6.4. Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

6.5. Vials - Glass, 2 ml capacity, with Teflon lined screw or crimp top.

6.6. Glass wool - contaminant free.

6.7. Disposable glass Pasteur pipet and bulb.

6.8. Apparatus for grinding.

6.9. Analytical balance - 0.01 g.

6.10. Test tubes

6.11. N-Evap concentrator with nitrogen source

6.12. Graduated pipets, 0.5, 1, 2 and 5 mL. Pipets are pre-tested by lot for accuracy.

7. REAGENTS

7.1. Pesticide grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One of SW-846.

7.3. Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours.

7.4. Extraction Solvents - Samples are extracted using one of the following solvent systems:

7.4.1. Acetone/Hexane (1:1) (v/v), CH_3COCH_3/C_6H_{14}. Pesticide quality or equivalent. Soil, sediment, and aqueous sludge samples.

7.4.2. Dichloromethane/Acetone (1:1 v/v), CH_2Cl_2/CH_3COCH_3. Pesticide quality or equivalent. Soil, sediment, and aqueous sludge samples.

7.4.3. Dichloromethane (DCM), CH_2Cl_2. Pesticide quality or equivalent. Other miscellaneous sample matrices.

8. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1. Refer to the applicable section in the determinative SOP for sample collection, preservation, and holding times.

8.2. The extract holding time is 40 days from sample preparation to analysis.

9. PREVENTIVE MAINTENANCE

9.1. Routine cleaning of the extraction apparatus is necessary, including all parts exposed to contact with samples, especially extraction thimbles and the Soxtherm apparatus.

9.2. The operating temperature of the soxtherm is monitored every quarter (March, June, September, and December) with a NIST traceable digital thermometer and probe. The soxtherm temperature is set at the controller to heat up to the extraction temperature of 140° C. Once the control box reads 140° C the probe is placed directly on each individual heating surface and the temperature is recorded in the soxtherm maintenance logbook (SVMP-SOX-01).

9.3. The extraction time on the controller is verified quarterly by recording the start and stop times of an extraction and recording in the soxtherm maintenance logbook.

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

11. PROCEDURE

11.1. Record all extraction and sample information on the applicable benchsheet. To assist the analyst, a brief description of the procedure is given on the backside of the benchsheet. See Attachment A.

11.2. Sample Handling

11.2.1. Sediment/soil samples - Mix the sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.2. Waste samples - Samples consisting of multi phases have each phase extracted separately. If sample contains a significant portion of water, water is pulled off and extracted. Oil phases are pulled off, usually diluted in hexane or iso-octane and extracted. This procedure is for solids only.

11.2.3. Dry waste samples amenable to grinding - Grind or otherwise subdivide the waste so that it passes through a 1 mm sieve.

11.2.4. Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling these difficult matrices.

11.2.5. Tissue samples need to be ground or cut to allow mixing and improve exposure of the sample to the solvent.

11.3. Determinations of sample % dry weight - In certain cases, sample results are desired based on dry weight basis. Refer to the *SOP for Determination of Percent Solids* (MET-160.3). If the determination is performed by the organics preparation personnel, a portion of the sample for this determination should be weighed out at the same time as the portion used for analytical determination.

11.4. Sample extraction and extract concentration

11.4.1. Pre-rinse glassware (beaker, thimble with glass wool, and metal clip) before use. To clean the glassware place 50mls of dichloromethane in beaker and run a short extraction program. Example 10 min. boil time, 1 reduction A, 10 min. extraction, 1 reduction B, 30 min. solvent cooling, temp. 140° C.

11.4.2. Refer to the determinative SOP (see Table 1 for a list of applicable SOPs) for the preparation, concentration, storage, and expiration for the surrogate, LCS, and MS spiking solutions. These SOPs also list the resulting final spike concentrations.

11.4.3. Weigh out the specified amount (see test-specific benchsheets attached) of the solid sample to be extracted, along with MB (anhydrous sodium sulfate) LCS (clean sand or sodium sulfate) samples. Record the sample weight on the benchsheet.

11.4.4. Blend the specified sample amount with as little sulfate as possible, so as not to overload the extraction thimble, but still achieve sample drying. Transfer the dry sample into the thimble that contains a plug of glass wool to prevent the sample from dropping into the beaker. Use a second thimble if the to prevent overfilling or over packing the thimble.

Note: Be careful not to weigh out more sample than what can fit into a maximum of 2 thimbles.

11.4.5. Add the surrogate standard spiking solution onto the samples. For the sample(s) in each analytical batch selected for QC spiking (LCS and MS samples), add the appropriate volume of the appropriate spiking standard. Each standard should be brought to room temperature before using. Addition of surrogate and spike is routinely witnessed by a second analyst to assure completeness. Also, the witness

should immediately follow the spiking of each sample with the addition of a small amount of dichloromethane.

11.4.6. Place extraction thimble in beaker such that the top of the thimble is flush or below the beaker mouth.

11.4.7. Add enough dichloromethane or appropriate extraction solvent to cover each sample. This will allow sample to remain covered by solvent during the entire boiling step. If the samples do not equally fill the thimbles feel free to mix and match sample and dichloromethane amounts so that all samples on a single extraction unit have about the same solvent level. Each unit can run a separate program, but a single unit does not allow multiple programs. In the program this step is referred to as boiling time.

11.4.8. The boiling step is set to be an hour. During this time the solvent will boil through and over the sample. The majority of the extraction will occur in this step. The temperature of the hot plates should be set for 140°C for dichloromethane and DCM/ACE. When the boiling time is completed the soxtherm needs to be programmed for the proper number of solvent reductions. Solvent reductions happen in 15mL increments as the calibrated cup is filled by the condensing solvent. When programming the number of reductions the original amount of solvent needs to be taken into consideration. The aim of this step is to bring the solvent level below the tip of the thimble with 50mls of being the desired amount. In the soxtherm program this is referred to as solvent reduction A.

11.4.9. Once the solvent level is below the thimble the extraction time can start. This lasts 1 hour. During this step the solvent drips through the sample to rinse through any remaining analytes. When this is done reduction B can begin. This step should take the solvent level to the desired amount of 5-15mL. Care should be taken during this step, since each beaker seals differently. Some beakers seal very well and others allow more solvent losses. Also all samples are not created equal. Some samples will trap more solvent than others.

11.4.10. If the solvent level has reached the desired stopping point before the programmed numbers of reductions are completed, it is possible to delete the remaining number and go right to the cooling step. Conversely, if the solvent level of the samples on an instrument is higher than desired extra solvent reductions can be programmed. Once the desired solvent amount has been reached cooling can begin. During this time the heating plates will cool down. This should be set for no less than 30 minutes to allow the hot plates to cool. It can be set for longer periods if desired.

11.4.11. Nitrogen blowdown is used to further concentrate the extract and to adjust the extract to the final volume. If solvent exchange is required (e.g. GC-ECD analyses) it is done at this point.

11.4.11.1. Using the N-Evap, place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

11.4.11.2. The internal wall of the tube must be rinsed down several times with the appropriate final solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). The volume of extract in the tube must be monitored during blowdown to avoid loss of more volatile analytes. Under normal operating conditions, the extract should not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

11.4.11.3. Adjust the extract final volume to the prescribed volume with the solvent last used. Measure the final extract volume using a 0.5, 1, or 2 mL graduated pipet, depending on the test.

11.4.12. If extract cleanup is to be performed, concentrate the extract to the appropriate volume. Proceed to the applicable cleanup procedure and SOP.

11.4.13. During the final volume step, transfer the extracts to an appropriate storage or autosampler vial. Label the vial with the sample or QC identification and store in the extract storage area. The extracts obtained may now be analyzed for the target analytes using the appropriate determinative technique. The extract holding time is 40 days from sample preparation to analysis.

12. QUALITY CONTROL

12.1. Refer to the SOP for the determinative method and *SOP for Sample Batches* for minimum QC requirements. Project-specific batching protocols may also be required.

12.2. The QC solutions required by the method must be added as described in the analytical method. The amount and identification of QC solutions added must be documented on the bench sheet. Any reagent blanks, laboratory control samples, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

13. DATA REPORTING AND REVIEW

13.1. Preparation of all samples must be documented on a bench sheet. All information regarding the sample(s) extracted, aliquoting, QC spiking, extraction steps, etc. must be documented by the person(s) performing the extraction.

13.2. The bench sheet must be reviewed by the extraction lead, supervisor, or instrument lab analyst. The instrument lab analyst should sign-off on the bench sheet, thus accepting custody of the extracts.

14. CORRECTIVE ACTION

14.1. Refer to the *SOP for Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis c) reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in the determinative procedures.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

17.3. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Perform an initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file.

18.2. Training is documented following the SOP *for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update II, September 1994, Method 3541, Revision 0.

19.2. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3500B, Revision 2

20. CHANGES SINCE THE LAST REVISION

20.1. Changed methylene chloride to dichloromethane throughout document.
20.2. Changed temperature in sec 11.4.1 from 120 to 140 C
20.3. Sec 3.3 is new.
20.4. Sec 9.2 and 9.3 are new.

TABLE 1

APPLICABLE DETERMINATIVE SOPs

POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS – LOW LEVEL PROCEDURE	SOC-8270L
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS	SOC-8082A

Attachments

Test-Specific Bench Sheets

Additional Prep Information For EPA 3541

Service Request _____ **Workgroup** _____

Sulfate Lot #_____ DCM ($GC^{2)}$ Lot #_____

Soxtherm Start (Time/Date/Initial): _____

Soxtherm Stop (Time/Date/Initial): _____

GPC Clean-up (3640): _____ (Initial/Date)

S-Evap Temp: _____

N-Evap Temp: _____

Hexane Exchange for Silica Gel: _____ Hexane Lot #_____

Silica Gel Clean-up (3630): _____ (Initial/Date)

Silica Column Lot #_____ 1:1 Hexane/DCM Reagent Lot #_____

Extract Storage: _____

Date Completed: _____

Comments/Observations:

_____

_____

_____

_____

_____

Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

COLUMBIA ANALYTICAL SERVICES, INC.

Appendix from EXT-3541

for Extracting Compounds in Soil or Tissue by

EPA Method 3541

Procedure:

Note: a) All anhydrous sodium sulfate (Na_2SO_4) used in this procedure must be thoroughly rinsed with methylene chloride (DCM).

b) Tissue samples require a pre-prep homogenizing procedure before extraction.

1. For low level work follow the low level glassware cleaning procedure.
2. Run the rinse cycle on Soxtherms prior to sample extraction. See EXT-3541 SOP and Operation Manual for programing instructions.

Prog. #01, Temp. limit=200°, Ext. temp.=120°, Ext time=10m, Rec. A=15 (5mL), Ext. time=10m, Rec. B=15 (5mL), std. cool=30m, Rec. rnse=5m, Rec pulse=3x, time=10m

3. All lab equipment that will have contact with the sample must be thoroughly rinsed with the extraction solvent.
4. Thoroughly mix sample. Decant water, remove large rocks, sticks, leaves and other organic matter unless this is the primary make-up of the sample. Weigh approximately *initial weight* grams wet weight into a 150ml beaker. Record Weight to the nearest 0.1 gram.
5. Weigh an equal amount of granular anhydrous Na_2SO_4[5] for the MB and LCS(s). Sediments require dup LCS's. For sediments and tissues weigh 5 grams of the appropriate SRM for analysis.
6. Mix sample with anhydrous Na_2SO_4[2] until sample is sandy and free flowing. Be carefil with amount of sulfate used so as not to overload thimble capacity. Multiple thimbles may be used.
7. Transfer dried extract to assembled Soxtherm thimble. Carefil not to contaminate rinsed labware.
8. Add surrogate and matrix spikes. See chart. **(Witnessed by a trained analyst)**
9. Add enough DCM to cover samples completely.
10. Place sample on Soxtherm and run the associated program. See EXT-3541 SOP and Operation Manual for programing instructions.

Prog. #03, Temp. limit=200°, Ext. temp.=120°, Ext time=1h, Rec. A=60 (15mL), Ext. time=1h, Rec. B=15 (15mL), std. cool=30m, Rec. rnse=5m, Rec pulse=3x, time=10m

11. Be carefil that the extract does not go dry. The goal is a volume of 10 to 20 mL after reductions. Samples are removed and immediately covered in foil as soon as this level is reached to allow other sample to continue the reduction step. Additional reduction may be required.
12. Quantitatively transfer the extract to culture tubes.
13. Based on sample analysis and compound list determine cleanup direction to take. See chart.
14. If a GPC cleanup is performed, only half the original extract is returned. Therefore the extract must be concentrated to 1/2 the final volume to achieve the correct "calculated" final volume.
15. Concentrate the extracts to *calculated final volume (see below)* on the N-Evap under a **gentle** stream of nitrogen with the temperature not exceeding 35°. Extreme caution must be used to ensure that the sample does not concentrate too low or to dryness.
16. Bring extracts to *calculated final volume (see below)* in DCM and place in a labeled, *colored (see below)*, 2mL autosampler vial.
18. Complete all necessary paperwork. Turn extracts and paperwork over to MS analysts.

Test	Initial Weight	Surrogate/ Amount	Spike/ Amount	Cleanup	Vial Color	Calculated Final Volume
8270-LL	20 grams dry	AP/50 µL	8270/50 µL + Benzoic Acid/50 µL	GPC	amber	2 mL
8270	30 grams dry	AP/1 mL	8270/1 mL	GPC	clear	1 mL
8270 Paperboards	20 grams	AP/1 mL	8270/1 mL + Benzidine/100 µl + Paperboard/20 µL	GPC	clear	1 mL
8270 Wipe	1 wipe	AP/1 mL	8270/1 mL	GPC	clear	1 mL
SIM-PAH/ SIM-ALK	10 grams dry	AP/20 µL	PAH/200 µl	Silica gel*	green	10 mL
SIM-PAH-PCP	10 grams dry	AP/20 µL	PAH/200 µL + Penta/400 µL	GPC	green	10 mL
SIM-PAH-UL Solid	20 grams dry	AP/20 µL	PAH/80 µL	Silica gel*	green	2 mL
SIM-PAH Paperboards	10 grams	AP/20 µL	PAH/200 µL	Silica gel*	green	10 mL
SIM-PAH Wipe	1 wipe	AP/10 µL	PAH/20 µL	Silica gel*	green	1 mL
SIM-PAH-UL Tissue**	10 grams	AP/20 µL **	PAH/100 µL **	Silica gel*	green	1 mL
SVO-SIM Tissue	5 grams	AP/50 µL	8270/50 µL	GPC	amber	10 mL
PBDE Paperboards	1 grams	PBDE/100 µL	PBDE/100 µL	Silica gel/ Sulfuric acid	amber	10 mL
PBDE Soil	10 grams dry	PBDE/50 µL	PBDE/50 µL	Silica gel/ Sulfuric acid	amber	2 mL
PBDE Tissue	10 grams	PBDE/50 µL	PBDE/50 µL	Silica gel/ Sulfuric acid	amber	2 mL
1653 Solid/ Paperboards	10 grams dry	1653 Labeled Std: Acetone/1 mL + Methanol/1 mL	1653 Secondary Std: Acetone/500 µL + Methanol/500 µL	Extract requires 1653 H2O extraction and derivitization	amber	0.5 mL
85.02 RA/FA Solid	10 grams dry	85.02/100 uL	85.02/100 uL + Retene/400 uL	Extract requires derivitization	amber	0.5 mL
OP Pest SVM/GCMS Solid	10 grams dry	SVM OP/100 uL	SVM OP/100 uL	GPC	amber	2 mL in 50/50 Hexane/Acetone
Pest/Pest	10 grams dry	SVM/100 uL	SVM/100 uL	Carbon	amber	1 mL

**If carbazole is in the compound list do not silica gel, perform GPC clean-up.*

***Add 100 µL of corn oil to the LCS, DLCS, and MB for SIM-PAH-UL Tissues.*

STANDARD OPERATING PROCEDURE FOR EXTRACTION OF

ORGANOCHLORINE PESTICIDES, PCB-AR, AND PCB CONGENERS IN SOILS

EPA 8081A / 8082-AR / 8082-CON by 3541

. Mix the sample thoroughly. Weigh appropriate amount into a beaker. Record the weight to the nearest 0.01 gram. Weigh out the same amount of muffled matrix sand for the lab control sample and muffled granular sodium sulfate for the method blank.

. Mix the samples with a sufficient amount of muffled sodium sulfate to completely dry the sample.

. Transfer samples into the Soxtherm thimbles.

. Add surrogates and analyte spikes as necessary.

. Add 150mL of DCM to the extraction beaker.

. Take extract to a 10 mL intermediate volume in DCM.

. Do **GPC cleanup (3640).** Refer to CAS SOP for GPC clean-up.

. Evaporate on the S-Evap, keeping temperature 70-75°C. When extracts are at about 10 mL, squirt about 5 mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (75°C) into hexane for until solvent stops dripping. Do not let the samples go dry.

. Concentrate extracts and do additional cleanups as needed.

10. Place pesticide extracts in a yellow vial, and PCB extracts in a green vial.

11. For Congener analysis, place exactly 500uL of extract into a green vial.

Test	Amount to weigh	Surr Amount	Spike/Amount	FV
8081	*10g*	*500uL*	*250uL*	*10mL*
8082	*10g*	*500uL*	*250uL*	*10mL*
8081-L	*20g*	*200uL*	*100uL*	*4mL*
8082-L	*20g*	*200uL*	*100uL*	*4mL*
8082-CON	*20g*	*CON/200uL*	*CON/200uL*	*4mL*

STANDARD OPERATING PROCEDURE FOR EXTRACTION OF

ORGANOCHLORINE PESTICIDES, PCB-AR, AND PCB CONGENERS IN SOILS

EPA 8081A / 8082-AR / 8082-CON by 3541

1. Mix the sample thoroughly. Weigh appropriate amount into a beaker. Record the weight to the nearest 0.01 gram. Weigh out the same amount of muffled matrix sand for the lab control sample and muffled granular sodium sulfate for the method blank.
2. Mix the samples with a sufficient amount of muffled sodium sulfate to completely dry the sample.
3. Transfer samples into the Soxtherm thimbles.
4. Add surrogates and analyte spikes as necessary.
5. Add 150mL of DCM to the extraction beaker.
6. Take extract to a 10 mL intermediate volume in DCM.
7. Do **GPC cleanup (3640).** Refer to CAS SOP for GPC clean-up.
8. Evaporate on the S-Evap, keeping temperature 70-75°C. When extracts are at about 10 mL, squirt about 5 mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (75°C) into hexane for until solvent stops dripping. Do not let the samples go dry.
9. Concentrate extracts and do additional cleanups as needed.
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11. For Congener analysis, place exactly 500uL of extract into a green vial.

Test	Amount to weigh	Surr Amount	Spike/Amount	FV
8081	*10g*	*500uL*	*250uL*	*10mL*
8082	*10g*	*500uL*	*250uL*	*10mL*
8081-L	*20g*	*200uL*	*100uL*	*4mL*
8082-L	*20g*	*200uL*	*100uL*	*4mL*
8082-CON	*20g*	*CON/200uL*	*CON/200uL*	*4mL*

STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM
Revision 6
October 18, 2007

Approved By:

Supervisor		10/18/07 Date
QA Manager		10-18-07 Date
Laboratory Manager		10/18/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: ___ Date: 10/1/08
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-82, modified for soil and sediment matrices (Puget Sound Estuary Program and Lloyd Kahn). Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. DEFINITIONS

3.1. Analysis Batch - Samples are analyzed in a set referred to as an analysis batch. The batch begins with calibration/standardization followed by QC analyses and samples. The batch ends when the QC analyses and set of samples has been completed.

3.2. Method Blank - The method blank is an artificial sample (empty boat) designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3. Laboratory Control Sample (LCS) - A standard of known TOC concentration which is used to ensure that the analysis produces an accurate measurement of TOC in samples analyzed in the batch.

4. INTERFERENCES

4.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

4.2. Volatile organics may be lost in the decarbonization process.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

5.5. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4±2°C. Samples are analyzed within 28 days of collection.

7. APPARATUS AND EQUIPMENT

7.1. Induction furnace, Coulometrics Incorporated.

7.2. Analytical balance, 0.1mg accuracy.

7.3. Desiccator.

7.4. Quartz combustion boats.

7.5. Sample scoop.

7.6. Porcelain dishes.

7.7. Glass ladles and miscellaneous laboratory glassware,

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Standards

8.1.1. Urea - 20% carbon. Use 10 μg.

8.1.2. Nutrients in Soil, purchased standard with a known TOC value (typically ERA #542). Use 50 mg for LCS.

8.2. Reagents

8.2.1. Hydrochloric acid, 50% and 10%.

8.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

8.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

8.2.4. Manganese dioxide. Gas scrubber solution.

8.2.5. Potassium Hydroxide. Gas scrubber solution.

8.2.6. Potassium Iodide. Anode chemical.

8.2.7. Magnesium Perchlorate desiccant

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the *SOP for Documentation of Training*, is also the responsibility of the department supervisor/manager.

10. PREVENTIVE MAINTENANCE

Maintenance is performed as follows:

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Precombustion Column	as needed
Repack Combustion Column	as needed

11. PROCEDURE

11.1. Sample Preparation.

11.1.1. Turn furnace on to #5 (≈1000°C). Allow furnace to warm-up for about 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

11.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

11.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

11.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10% HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

11.2. Apparatus Preparation.

11.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

11.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

11.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

11.2.4. Place cell in coulometer cell holder.

11.2.5. Turn on detector lamp and stir plate. (Power on)

11.2.6. Turn adjust knob to 122 (all the way to the right) then turn back down to 100. Rotate cell until maximum transmittance is obtained.

11.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

11.2.8. Change Magnesium Perchlorate desiccant daily.

11.2.9. The instrument is now ready to run.

11.3. Calibration and Standardization.

11.3.1. Burn both ladles for five minutes each to remove any residual TOC.

11.3.2. Establish baseline.

11.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

11.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

11.4. Analysis.

11.4.1. Place one platinum or quartz boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

11.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

11.4.3. Load the other ladle with the next platinum (or quartz) boat. Remove the ladle in use from the inlet port and insert the next ladle.

11.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

The precision and accuracy of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS's are prepared and analyzed. The RSD should be $<20\%$ and average recovery must be 85-115%.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Analyze a minimum of seven spiked blank replicates at a level near the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection*.

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described below and in the *SOP for Sample Batches*. Project-specific batching protocols may also be required.

12.3.1. LCS - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze 50mg of the purchased standard (see 8.1.2) is used. The acceptance criteria for this LCS is ± 15% of the true value.

12.3.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be <0.05% carbon.

12.3.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Analyze ~10mg urea. The CCV must be 18.0% - 22.0% carbon.

12.3.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

12.3.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

12.3.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

12.3.7. See Table 1 for a summary of acceptance criteria and corrective actions.

13. DATA REDUCTION AND REPORTING

13.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ -\ baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

13.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

13.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

13.7. Reporting

13.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

13.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

13.7.3. Report all results to three significant figures.

13.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method is validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

17.3. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. REFERENCES

18.1. Coulometrics Inc. Instruction Manual, Model 5020.

18.2. EPA Method Modified 415.1.

18.3. Total Organic Carbon, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0.

18.4. Total Organic Carbon(TOC), Conventional Sediment Variables, Puget Sound Estuary Program, March 1986.

18.5. Determination of Total Organic Carbon in Sediment, Lloyd and Kahn, U.S.E.P.A Region II, July 1998.

18.6. ASTM Method D4129-88

TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

APPENDIX I

BENCHSHEETS

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes TOC % = $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

CCV1 =97 CCV2 =

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C REC%=

Comments :

X= RSD= SPK%REC=

Analyzed By:	Date:
Reveiwed By:	Date:

TOC.XLT

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes TOC % = $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C % Rec =

Comments :

Analyzed By:	Date:
Reveiwed By:	Date:

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
				AVG.

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
CCV-1				
CCB-1				
LCS				
MB				Avg
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

Columbia Analytical Services, Inc.

Service Request #: K7112 / K7040 / K7669
Method: 9060s / PSEP (Combustion/Coulometric)
Analysis For: Total Organic Carbon (TOC)
Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon
CCV-1	6.8	1328.3	7.3	1321.0	19.4
CCB-1	50.0	6.8	7.3	-0.5	< 0.05
LCS-1	47.4	198.4	7.3	191.1	0.40
MB-1	50.0	7.2	7.3	-0.1	< 0.05
LCS-2	47.8	201.1	7.3	193.8	0.41
LCS-3	56.1	247.8	7.3	240.5	0.43
LCS-4	46.1	238.5	7.3	231.2	0.50
LCS-5	47.5	196.5	7.3	189.2	0.40
LCS-6	46.2	194.2	7.3	186.9	0.40
LCS-7	47.0	200.7	7.3	193.4	0.41
LCS-8	46.0	214.9	7.3	207.6	0.45
LCS-9	47.1	194.8	7.3	187.5	0.40
LCS-10	45.6	203.5	7.3	196.2	0.43
CCV2	10.7	2074.1	7.3	2066.8	19.3
CCB-2	50.0	9.3	7.3	2.0	< 0.05

Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** (Net Reading)(µg 0.1) / mg Sample Injected

CCV : Urea Baker (lot #A17584) ID#: TOCS/1-10-J TV = 20.0%C

CCV1 = 97 CCV2 = 97

LCS: ERA Cat#: 542 Lot#: D060-542 ID#:PO3/2-26E TV = 0.34%C

Comments :

LCS 1 to 10 $\bar{x}$ = 0.42 RSD = 5%

ERA QC Performance Acceptance Limits for TOC are 436 to 6370 mg/kg

MATERIAL STANDARDIZED TO 0.42%. USE AS TV FOR THIS LOT CWF 8-27-08

Analyzed By:		Date: 8/26/08	Time: 0900
Reveiwed By:		Date: 8/28/08	

TOC.XLT

STANDARD OPERATING PROCEDURE

PARTICLE SIZE DETERMINATION – ASTM PROCEDURE

GEN-PSASTM
Revision 1
November 20, 2007

Approved By:

Supervisor	11/20/07 Date
QA Manager	11-20-07 Date
Laboratory Manager	11/20/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

PARTICLE SIZE DETERMINATION – ASTM PROCEDURE

1. SCOPE AND APPLICATION

1.1. This procedure covers the quantitative determination of the distribution of particle sizes in soils as described in ASTM D 422-63. The distribution of particle sizes larger than 75 μm is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to obtain the necessary data.

1.2. Detection limits are determined from accuracy of analytical balances. Samples are weighed to the nearest 0.01g and results are reported to the nearest 0.01 percent.

1.3. The pre-preparation of the samples employs ASTM Method D421-85.

2. METHOD SUMMARY

Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological variables, it can be used to normalize chemical concentrations according to sediment characteristics and to account for some of the variability found in biological assemblages. Particle size is also an important variable for marine engineering purposes.

3. DEFINITIONS

3.1. Particle size – The size of various solid components making up a sediment, as named in the applicable method reference (gravel, sand, silt, clay, etc.).

3.2. Dispersing agent – A solution introduced into the soil suspension to disperse the small sediment fractions and reduce flocculation, allowing more accurate determination of particle sizes using a hydrometer.

4. INTERFERENCES

Depending on the required particle size distribution, organic material can be an interference.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Samples can be collected in glass or plastic containers. A minimum sample size of 300-2000g is recommended. If unrepresentative material is to be removed from the sample, it should be removed in the field under the supervision of the chief scientist and noted on the field log sheet.

6.2. Samples should be stored at 4 ± 2 °C, and can be held for up to 6 months before analysis. Samples must not be frozen or dried prior to analysis, as either process may change the particle size distribution.

7. APPARATUS AND EQUIPMENT

7.1. Sieve shaker - Ro-Tap or equivalent

7.2. Drying oven

7.3. Mortar and rubber covered pestle

7.4. Thermometer accurate to 0.5 °C

7.5. Analytical balance - 0.1mg accuracy

7.6. Desiccator

7.7. Clock - with second hand

7.8. Standard sieves - Appropriate mesh sizes, sieve pan and top, sieve brush.

7.9. Funnel

7.10. Graduated cylinders

7.11. 250-mL beakers

7.12. Hydrometer – An ASTM hydrometer, graduated to read specific gravity and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100.

7.13. Stirring apparatus as described in ASTM D422-63 Section 3.2.1.

7.14. Water pique or squirt bottle

7.15. Glossy paper

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Dispersant - 4 percent sodium hexamethaphosphate. To prepare, weigh 40.0g sodium hexamethaphosphate and dilute to 1.0L in DIW.

8.2. Distilled water

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed for sieves other than normal cleaning and inspection.

9.2. Balance calibration checks are performed daily.

9.3. Color-indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically, and, if necessary, the ground glass rims should be greased or the "0" rings should be replaced.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the *SOP for Documentation of Training* (ADM-TRANDOC).

11. PROCEDURE

11.1. Sample Preparation (from ASTM D421-85)

11.1.1. As received samples shall be air-dried at room temperature until thoroughly dried.

11.1.2. A portion of the as received sample shall be used to determine the specific gravity of the sample for later use on the report.

11.1.3. Air-dried samples shall be broken up into the individual aggregates using a mortar and rubber covered pestle. Analyst will take care not to reduce the size of individual particles, but only de-aggregate clumps of dried material.

11.1.4. After de-aggregation, select approximately 10-15g for hygroscopic moisture content. Moisture content is to be measured by drying the sub sample at 110 ± 5 °C.

11.2. Analysis

11.2.1. Select a portion of the air-dried sample for the purpose of testing. Separate the test sample by sieving with a No. 10 sieve. Grind the fraction retained on the No. 10 sieve with a mortar and rubber covered pestle until the aggregations of soil particles are broken up into separate grains and re-sieve that portion.

11.2.2. Wash the fraction retained on the No. 10 sieve free of all fine material, dry at 110 ± 5 °C, and weigh. Record this mass as the mass of course material.

11.2.3. Separate the portion retained on the No. 10 sieve into a series of fractions using the 3/4, 3/8 in, No. 4 (4.75 mm) and No. 10 (2.00 mm) sieves and a Ro-Tap shaker.

11.2.4. Determine the mass of each fraction using a balance accurate to 0.1mg and record on the benchsheet.

11.2.5. Thoroughly mix together the fractions passing the No. 10 sieve in both sieving operations, and by the method of quartering, select a portion weighing approximately 115g for sandy soils and 65g for silt and clay soils.

11.2.6. Take 10 to 15g of the sample passing the No. 10 sieve, dry it at 110 ± 5 °C, and record the weight. Enter these weights on the benchsheet under hygroscopic moisture.

11.2.7. Weight a portion of the remaining sample passing the No. 10 sieve. Use approximately 50g when the sample is mostly silt and/or clay and approximately 100g when the sample is mostly sand. Place this portion in a 250 mL beaker and cover with 125 mL of sodium hexametaphosphate solution and stir until thoroughly wetted. Allow to soak for at least 16 hours.

11.2.8. After the 16 hour soaking period, move wetted sample into a stirring apparatus as described in ASTM D422-63 Section 3.2.1. Stir sample for a period of one minute and transfer slurry into a sedimentation cylinder. Bring the final volume up to 1 L.

11.2.9. Determine the correction factor for each hydrometer by placing 125 mL sodium hexametaphosphate solution in a sedimentation cylinder, and diluting to 1 L with de-ionized water. Place the hydrometers in the cylinder and take a reading. This number will be subtracted from each reading taken. It is important to note that the readings should be taken at the top of the meniscus as it is nearly impossible to see the bottom of the meniscus when soil particles are present in the suspension.

11.2.10. Cap the sedimentation cylinder with Parafilm and turn the cylinder upside down and back for a period of one minute. At the end of the one minute, set the cylinder on the counter, uncap, and take hydrometer readings of the suspension at the following intervals: 2, 5, 15, 30, 60, 250, and1440 minutes. When a hydrometer reading is taken, carefully insert the hydrometer into the suspension 20 to 25 seconds before the desired reading to allow the hydrometer to settle. After the reading is taken, carefully remove the hydrometer from the suspension and place it with a spinning motion in a graduate of clean de-ionized water. Record the temperature of the suspension after each hydrometer reading.

11.2.11. After the last hydrometer reading is taken, transfer the suspension to a No. 200 (75 μm) sieve and wash with tap water until the wash water is clear. Transfer the material to a suitable weighed container and dry to a constant weight in an oven at110 ± 5 °C, record dried sample weight, and make a sieve analysis of the portion retained using the No. 20, No. 40, No. 60, No. 140, and No. 200 sieves and a pan.

12. QA/QC REQUIREMENTS

For ASTM D422-63 one duplicate analyses shall be conducted on one of every 20 samples, or one sample per batch if less than 20 samples are analyzed.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. The data is entered into a spreadsheet and results determined using the appropriate equations. Refer to Appendix A.

13.2. Reporting and review

13.2.1. The weight of each sediment fraction should be reported to the nearest 0.0001g dry weight. The laboratory should report the results of all samples analyzed (including QA replicates) and should note any problems that may have influenced data quality.

13.2.2. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified for samples (above). These results are then used to calculate QC determinations

13.2.3. The results are entered directly onto the appropriate EDD forms located in the CAS network directory R:\WET\WIP. Refer to Appendix A. Once the results are transferred, the data and report are reviewed.

13.2.4. Refer to the SOP for Laboratory Data Review Process for general instructions for data review.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

Refer to the reference method for method performance data available.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the

volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Independently perform the analyses. For Initial Demonstration of Capability the data must be reviewed by a supervisor and the supervisor must document that the analyst is trained.

18.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

ASTM Procedure D422.

Appendix A

Benchsheets and Spreadsheets

Columbia Analytical Services, Inc.

Sample Name :	sample 1	Service Request:	0
Lab Code:	-001	Method:	D421 / D422
Client:	0	Date Collected:	01/00/00
Project:	0	Date Received:	01/00/00
Sample Matrix:	0	Date Analyzed:	01/00/00
Time Started:	0000		

Sample Preparation (ASTM D421-85)

(1) Mass of total test sample. (g) (6.1) _____

(2) Coarse Material (6.2)

Coarse material + Tare. (g) _____

Tare weight. (g) _____

(3) Mass retained on the No. 4 sieve. (g) (6.2) _____

(4) Selected portion of fine material. (g) (7.1) _____

Particle Analysis (ASTM D422-63)

Sieve Analysis Of Coarse Material. (6.1)

Gravel 19.0 mm	3/4"	
Gravel 9.50 mm	3/8"	
Gravel 4.75 mm	4	
Gravel 2.00 mm	10	

Sieve Analysis Of Fine Material. (11.1)

Gravel 0.850 mm	20	
Gravel 0.425 mm	40	
Gravel 0.250 mm	60	
Gravel 0.106 mm	140	
Gravel 0.075 mm	200	
Pan	-	

Analysis of fine material.

Hydrometer corrections. (7.3)

	Hydro 1	Hydro 2
Reading:		

Hygroscopic moisture. (8.1)

Air dried (g)	
Tare (g)	
Oven dried + Tare	

Weight of Fine material

Dry wt.+Tare (g)	
Tare (g)	
Fine material (g)	

Determination of Silt/Clay Fraction. (10.2) (10.3) (10.4)

(10.2) T (Min)	(10.2) Hydro Reading	(7.3) Corr. Fact.	(10.4) Temp °C	Specific Gravity		Table 1 a		Table 3 K		
2										
5										
15										
30										
60										
250										
1440										

Analyst : _____ Date : _____

Reviewed by : _____ Date : _____

Client:	0	**Service Request:**	
Project:	0	**Date Collected:**	
Sample Matrix:	0	**Date Received:**	
		Date Analyzed:	

ASTM Method D422 Particle Size

Sample Name: sample 1 **Lab Code:** -001

Initial Weight of air-dried sample. **(6.1)**	
Mass retained on the No. 4 sieve. **(6.2)**	
Air-dried portion of fine material. **(7.1)**	

Course Sieving ASTM D421 (6.2)

Mass of coarse material (g)	
Percent Recovered	

Coarse sieving (6.1)		**Sieve #**	**Weight (g)**	**% Passing**	**Mass Passing**
Gravel	19.0 mm	3/4"			
Gravel	9.50 mm	3/8"			
Medium Gravel	4.75 mm	4			
Fine Gravel	2.00 mm	10			
Fine sieving (11.1)					
V.C. Sand	0.850 mm	20			
C. Sand	0.425 mm	40			
M. Sand	0.250 mm	60			
F. Sand	0.106 mm	140			
V.F. Sand	0.075 mm	200			**(16.2)**

Calculated amount retained on the # 10 sieve. **(16.1)**	

Total Recovered (g)	
Total Recovered (%)	

Hygroscopic Moisture (8.1)

Air-dried sample (g)	
Oven dried sample (g)	
Hygroscopic Moisture **(13.1)**	

(W) **(14.2)**	

Oven-Dried Sample used in hydrometer analysis. (g) **(14.1)**	

Determination of Silt/Clay Fraction

(10.2) T (Min)	**(10.2)** Hydro Reading	**(7.3)** Corr. Fact.	**(10.4)** Temp °C	**Table 1** Specific Gravity	H (Net)	**(14.3)** a	**Table 2** L Eff. Depth	**Table 3** K	**(14.3)** % Passing = H/Wa * 100	**(15.1)** Dia-meter
2										
5										
15										
30										
60										
250										
1440										

Comments:

Analyzed By:	Date:
Reviewed By:	Date:

Sieve	X arithmetic Percent Passing (%)	Y logarithmic Particle Diameter (mm)	Convert Y mm to nm (nm)	Value of Y Log form (log)
3/4"		19.0	19000000	7.279
3/8"		9.5	9500000	6.978
4		4.75	4750000	6.677
10		2.00	2000000	6.301
20		0.850	850000	5.929
40		0.425	425000	5.628
60		0.250	250000	5.398
140		0.106	106000	5.025
200		0.0750	75000	4.875
2			#VALUE!	#VALUE!
5			#VALUE!	#VALUE!
15			#VALUE!	#VALUE!
30			#VALUE!	#VALUE!
60			#VALUE!	#VALUE!
250			#VALUE!	#VALUE!
1440			#VALUE!	#VALUE!

determined hydrometer mm	mm to nm	log hyd x	% Passing
0.074	74000	4.87	#VALUE!
0.005	5000	3.70	#VALUE!
0.001	1000	3.00	#VALUE!

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: 0
Project: 0
Sample Matrix: 0

Service Request: 0
Date Collected:
Date Received:
Date Analyzed:

Particle Size Determination
ASTM Method D 422

Sample Name: sample 1
Lab Code: -001

Gravel and Sand
(Sieve Analysis)

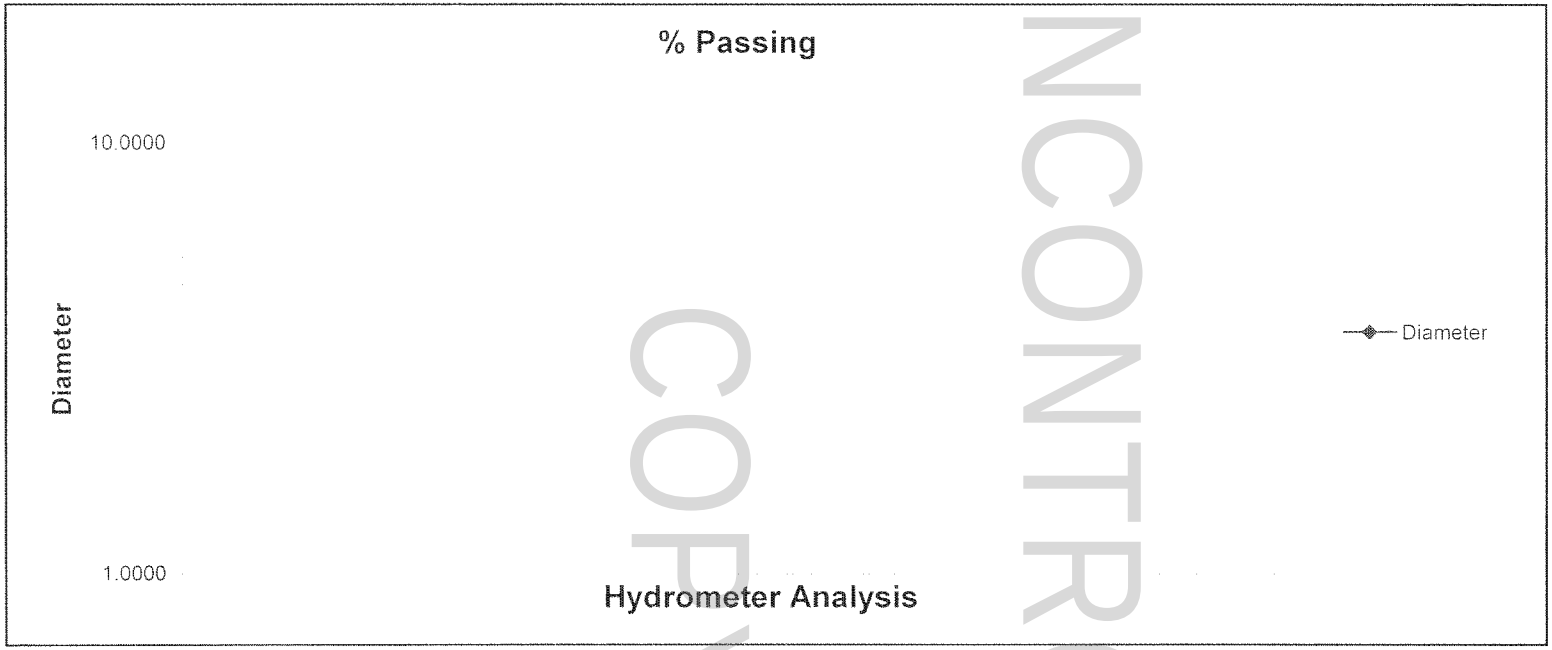
Description	Sieve Size	Weight (g)	Percent Passing
Gravel	No.3/4"(19.0 mm)		
Gravel	No.3/8"(9.50 mm)		
Gravel, Medium	No.4 (4.75 mm)		
Gravel, Fine	No.10 (2.00 mm)		
Sand, Very Coarse	No.20 (0.850 mm)		
Sand, Coarse	No.40 (0.425 mm)		
Sand, Medium	No.60 (0.250 mm)		
Sand, Fine	No.140 (0.106 mm)		
Sand, Very Fine	No.200 (0.0750 mm)		

Silt and Clay
(Hydrometer Analysis)

Particle Diameter	Percent Passing
0.074 mm	#VALUE!
0.005 mm	#VALUE!
0.001 mm	#VALUE!

Approved By: _____ Date: _____
1A/102094

Sample Name: sample 1
Lab Code: -001



STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS); EPA METHOD 6020

SOP No.: MET-6020

Revision: 14

April 10, 2010

Approved by: _____ Supervisor — 3/19/10 Date

_____ QA Manager — 3/19/10 Date

_____ Laboratory Manager — 3/19/10 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS) EPA METHOD 6020

1 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentrations of certain elements in water, soil, tissues, aqueous and non-aqueous wastes, and sediment samples using EPA Method 6020 or 6020A. Table 1 indicates analytes that are typically determined by this procedure and lists the standard Method Reporting Limits (MRLs) for each analyte in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. Project-specific MRLs may apply, and if lower than standard MRLs, it is demonstrated through method detection limit determinations and analysis of MRL standards that the MRL is achievable. Method Detection Limits (MDLs) that have been achieved are listed in Table 1. These may change as annual studies are performed.

1.2 The complexity of the technique generally requires outside study of appropriate literature as well as specialized training by a qualified spectroscopist. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

2 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be digested using appropriate sample preparation methods. The digestate is analyzed for the elements of interest using ICP-mass spectrometry (ICP-MS).

2.2 Methods 6020 and 6020A describe the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3 DEFINITIONS

3.1 **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2 **Independent Calibration Verification (ICV)** - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3 **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes is added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.4 **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.5 **Duplicate Sample** (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.6 **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire procedure.

3.7 **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8 **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

4 INTERFERENCES

4.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isoptope.

4.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could

affect ICP-MS determinations have been identified in the literature. Refer to Method 6020/A for further discussion.

5 SAFETY

5.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2 Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3 Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4 High Voltage - The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

5.5 UV Light - The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until digestion. Soil or solid samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until digestion.

6.2 Samples are prepared via procedures in SOPs MET-DIG, MET-3020A, or MET-3050 depending on matrix and project specifications.

6.3 Digestates are stored in the appropriate volumetric containers. Following analysis, digestates are stored until all results have been reviewed. Digestates are neutralized prior to disposal through the sewer system, 2 weeks after data is reviewed.

7 APPARATUS & EQUIPMENT

7.1 Instruments: Thermo Elemental ExCell (K-ICP-MS-02) Serial # EX191, and Thermo Elemental X-Series (K-ICP-MS-03) Serial # X0193.

7.2 Thermo Meinhard type (Part # 1201318)

7.3 Thermo Impact Bead Quartz Spray Chamber (Part # 3600170)

7.4 Thermo X7 Nickel Sample Cone (1.0 mm orifice) (Part # 3004661), or Xi sample cone (part # 3600812)

7.5 Thermo X7 Nickel Skimmer Cone (0.75 mm orifice) (Part # 3200860) or Xi skimmer cone(part # 3600811)

8 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 All standards are prepared from NIST traceable standards. The expiration dates are assigned according to the EPA method and the vendor's assigned expiration dates. For example, working ICS solutions are prepared weekly in accordance with Method 6020, Section 5.6.1.

8.1.1 1000 ppm Single Element Stock Standard Solutions: Each stock standard is store at room temperature on shelves located in room 113 of the metals lab. The manufacturer, lot number, and expiration date of each stock standard is recorded in a bound logbook also located in room 113. Additionally each stock standard is given a unique, identifying name.

8.1.2 Intermediate Standard Solutions: Intermediate mixed stock solutions are made from the individual stock standards described above. The individual component of each mixed solution is recorded in a bound logbook located in the ICP-MS laboratory and mixed solution is given a unique, identifying name. The expiration date for the intermediate standard is the earlier of any one of its stock components.

8.1.3 Calibration Standards: Calibration standards are made fresh daily from the intermediate standard solutions. Each individual intermediate standard used in the calibration standard is recorded in a bound logbook located in the ICP-MS laboratory, and the calibration standard solution is given a unique, identifying name. The calibration standards unique name is used on the raw data to link the data to the subsequent prepared standards and ultimately the original purchased stock standard.

8.2 Standards Preparation

8.2.1 Expiration of all standard solutions defaults to the earliest expiration date of an individual component unless otherwise specified.

8.2.2 Calibration Standards

The calibration standard is prepared from two intermediate stock solutions. These solutions are prepared in acid rinsed 1000 mL Class A volumetric flasks following the formulations laid out on the attached example standard sheet (see Attachments). The working calibration standard is made daily by aliquoting 2.5 mL of each of the intermediate solutions in to a 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This standard is also used as the Continuing Calibration Verification (CCV).

8.2.3 Initial Calibration Verification (ICV)

8.2.3.1 The ICV intermediate stock solution is prepared in an acid rinsed 100 mL Class A volumetric flask. The solution is prepared by adding 2.0 mL of Inorganic Ventures QCP-CICV-1, 1.0 mL each of QCP-CICV-2 and QCP-CICV-3, 0.5 mL of 1000 ppm Molybdenum stock solution, 0.5 mL of 1000 ppm Uranium stock solution and diluting to volume with 1% HNO3.

8.2.3.2 The working ICV solution is prepared by aliquoting 0.5 mL of the mixed ICV intermediate solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3.

NOTE: The ICV solution is not at the midpoint of the linear range which may be as high as 1000 µg/L for some elements. The ICV solution used is a premixed standard purchased from Inorganic Ventures and contains the elements of interest between 2.5 and 100 µg/L. This solution provides calibration confirmation at more representative levels, given that most ICP-MS analyses are quantifying analytes in the low-ppb to sub-ppb range.

8.2.4 Interference Check Solutions (ICSA and ICSAB)

8.2.4.1 The ICSA is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02) solution and diluting to volume with 1% HNO3.

8.2.4.2 The ICSAB is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02), 0.125 mL of Inorganic Ventures 6020ICS-9B, and 0.250 mL of 10 ppm Molybdenum solutions and diluting to volume with 1% HNO3.

8.2.5 Post-digestion spikes are performed by adding appropriate amounts of the calibration intermediate solutions to aliquots of the sample digestate. The volumes of each standard

used vary based on the native concentrations found in the field samples. Refer to the post-digestion spike in Section 12 for details.

8.2.6 Refer to the appropriate digestion SOP for details of LCSW and matrix spike solution composition and preparation.

8.2.7 Tuning / Mass Calibration Solution

8.2.7.1 A 10 ppm intermediate solution containing Ba, Be, Bi, Co, In, Li, Pb, Mg, Tl, and U is prepared by adding 10 mL of each from 1000 ppm stock standards to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. The expiration date for the intermediate solution is the earliest of any one of its stock components.

8.2.7.2 The working solution is prepared in two ways. For the ExCell (K-ICP-MS-02) a 1.0 ppb tune/mass calibration solution is prepared by adding 0.10 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. For the X-Series (K-ICP-MS-03) instrument a 5.0 ppb tune/mass calibration solution is prepared by adding 0.50 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. The expiration date for this solution is taken from the intermediate stock above.

8.3 Internal Standards Stock Solution – Prepare a 10 µg/mL solution containing ^{71}Ga, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh ^{45}Sc, and ^{89}Y by adding 10.0 mL of each 1000 ppm single element stock solution to a acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric. Use this solution for addition to blanks, calibration standards and samples at a ration of 0.5 mL of internal standard to 100 mL of solution, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump.

8.4 Additional Reagents

8.4.1 Reagent water, ASTM Type II

8.4.2 "OmniTrace Ultra" Concentrated Nitric Acid (EM Science # NX0408-2)

8.4.3 Argon (Airgas Industrial Grade – 99.999% pure, bulk delivered)

9 PREVENTIVE MAINTENANCE

9.1 All maintenance is documented in the instrument logbook. CAS/Kelso maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor.

9.2 Most routine maintenance and troubleshooting is performed by CAS staff. Preventive maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Other maintenance or repairs may, or may not require factory service, depending on the nature of the task.

- cone removal and cleaning
- removal and cleaning of ICP glassware and fittings
- checking and cleaning RF contact strips
- checking air filters and cleaning if necessary
- checking the oil mist filters and cleaning if necessary
- checking the rotary pump oil and adding or changing if necessary
- removal and cleaning of extraction lens
- removal and cleaning of ion lens stack
- replace the electron multiplier as necessary

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for Documentation of Training, is also the responsibility of the department supervisor/manager.

11 PROCEDURE

11.1 Refer to method 6020 (or 6020A) and the instrument manuals for detailed instruction on implementation of the following daily procedures preceding an analytical run.

11.2 After the instrument has been placed in the "Operate" mode, begin completing the daily instrument log (see Attachments). Refer to the instrument manuals for the optimum settings for each instrument.

11.3 The following parameters are monitored to assure awareness of changes in the instrumentation that serve as signals that optimum performance is not being achieved, or as indicators of the physical condition of certain consumable components (i.e. EMT and cones).

11.3.1 Multiplier Voltages

11.3.2 Gas Flows - Coolant Ar

11.3.3 The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.4 Optimization

11.4.1 Gas Flows

11.4.1.1 Allow a period of not less than 30 minutes for the instrument to warm up.

11.4.1.2 Aspirate a mixed tune solution into the plasma and monitor the instrument output signal of In at mass 115 on the ratemeter. Adjust the nebulizer and auxiliary flows to obtain maximum signal. Adjust the tension screw on the peristaltic pump to obtain minimum noise in the analytical signal. Record flow rates and note any large variances.

Note: Significant differences in flow rates will be observed for different torches and cones.

11.4.2 Tuning

11.4.2.1 Ion Lens Setting - While monitoring the output signal of a mixed tune solution at mass 115 on the ratemeter, adjust the ion lenses to obtain maximum sensitivity. Refer to the instrument manual for details on performing the adjustments.

11.4.2.2 Mass Calibration - Aspirate the tune / mass calibration solution described in section 9.2.7.2 and perform the mass calibration using the instrument's Mass Calibration program. (Refer to the instrument manual for details pertaining to the mass calibration procedure.) The acceptance criteria for the mass calibration is <0.1 amu from the true value. If the mass calibration fails criteria re-tune the instrument and perform the mass calibration procedure again.

11.4.2.3 Resolution Check - Using the spectra created during the mass calibration procedure; perform the resolution check to assure the resolution is less than 0.9 AMU at 10% peak height. If the resolution does not pass criteria adjust the instrument's resolution settings, run a new scan of the mass calibration solution and recheck.

11.4.2.4 Stability Check - Using the tune / mass calibration solution, perform a short-term stability check as per EPA Method 6020 or 6020A. The relative standard deviations of five scans for each element in the tune solution must be < 5%. If the test does not pass criteria determine the cause (i.e. dirty cones, improper tune, etc.) correct the problem and re-run the test.

11.5 Analytical Run

11.5.1 Calibrate the instrument using a calibration blank (Standard 0), composed of reagent water and 1% nitric acid, and the working calibration standard (8.2.2). The masses typically monitored and those used for quantification are listed in Table 1. These masses are set as defaults in the instrument's analytical procedures. To begin select the correct method. Nebulize Standard 0 (Blank) into the plasma. Allow 1-2 minutes for system to equilibrate prior to establishing baseline. Follow directions on computer screen to perform standardization. Nebulize the working calibration standard into the plasma. The operator must sign and date the first page of standardization.

11.5.2 After the first CCB and before the ICS standards a CRA (MRL / LLICV / LLCCV) standard is analyzed. Method 6020 requires the detection to be > the MDL but < 2x the MRL. For 6020A, the criteria are 70-130% recovery. For DoD projects, the CRA criteria are 80-120%.

Note: For 6020A the LLCCV must also be analyzed at the end on the analytical run sequence.

11.5.3 Perform the analysis in the order listed below. A daily run log of all samples analyzed is maintained.

Initial Calibration Verification (ICV)
Continuing Calibration Verification (CCV)
Initial Calibration Blank (ICB)
Continuing Calibration Blank (CCB)
CRA (MRL / LLICV / LLCCV)
ICSA
ICSAB
Analyze 10 Samples
CCV
CCB
Analyze 10 Samples
CCV
CCB
Repeat sequence as required to complete analytical run, analyzing CCVs/CCBs every 10 analyses and at the end of the run.

12 QA/QC REQUIREMENTS

12.1 Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four

LCS aliquots are prepared and analyzed. The average percent recovery of for each analyte must be 85-115% (for water, and within the LCS limits for soils) and the RSD <20%.

12.2 Method Detection Limits

12.2.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank matrices at a level near or below the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL)* details of performing the MDL study.

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDL's must be verified annually or whenever there is a significant change in the background or instrument response.

12.3 For method 6020A, an LLQC sample (a CRA that is carried through the digestion) must be analyzed to verify accuracy at the MRL. The recovery must be 70-130%.

12.4 Instrument Detection Limits (IDLs) and linear ranges studies are performed quarterly. These will be calculated and made available to the ICP-MS operator. Linear range studies determine the Linear Dynamic Range (LDR) of the each instrument by analysis of a high concentration standard with results with ± 10% of the expected value. For non-DoD projects samples may be quantified between the MRL and 90% of the LDR without flagging. The Linear Calibration Range (LCR) is established by the highest calibration standard.

- **Note:** IDLs must be < LOD for DOD projects. DoD project samples with concentrations above the calibration standard must be diluted to bring results within the quantitation range. The LOQ and cal standard establish the quantitation range. The lab may report a sample result above quantitation range if the lab runs and passes a CCV that is > sample result.

12.5 The Initial Calibration Verification (ICV) standard is analyzed immediately after calibration. The results of the ICV must agree within ±10% of the expected value. If the control limits are exceeded, the problem will be identified and the instrument recalibrated.

12.6 A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed after calibration then every 10 samples thereafter with a final CCV/CCB closing the final samples of the analytical run.

12.6.1 The results of the CCV must agree within ±10% of the expected value.

12.6.2 The CCB measured values must be less than the MRL / LOQ for each element for standard applications. Other project-specific criteria may apply (for DoD QSM projects CCB can have no analytes > the LOD).

12.6.3 If the control limits are exceeded, the problem will be identified and corrective action taken. The instrument recalibrated. The previous 10 samples must be reanalyzed.

12.7 The ICSA and ICSAB solutions are analyzed after calibration and before any field samples. The solutions are then reanalyzed every 12 hours. Results of the ICSA are used by the analyst to identify the impact of potential interferences on the quality of the data. Based on these results appropriate action should be taken when interferences are suspected in an field sample including, but not limited to, selecting and alternative isotope for quantification, manual correction of the data, elevating the MRL, selection of an alternative method (e.g. optical ICP, GFAA) or flagging the result as estimated when no other action is possible. Results for the spiked analytes in the ICSAB solution must agree with ± 20% of the expected value.

INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

	Solution A Concentrations (mg/L)	Solution B Concentrations (mg/L)
Al	20.0	20.0
Ca	60.0	60.0
Fe	50.0	50.0
Mg	20.0	20.0
Na	50.0	50.0
P	20.0	20.0
K	20.0	20.0
S	20.0	20.0
C	40.0	40.0
Cl	424	424
Mo	0.05	0.05
Ti	0.40	0.40
As	0.0	0.025
Cd	0.0	0.025
Cr	0.0	0.050
Co	0.0	0.050
Cu	0.0	0.050
Mn	0.0	0.050
Ni	0.0	0.050
Se	0.0	0.025
Ag	0.0	0.0125
V	0.0	0.050
Zn	0.0	0.025

NOTE: The concentration of interfering elements in the ICSA and ICSAB solutions are spiked at levels 5 times lower than recommended in Table 1 of Method 6020A. Running the full

strength solutions as described in 6020A introduces too much material approximately 0.35 % dissolved solids into the ICP-MS system when trying to conduct low level analysis. Since the ICP-MS instrumentation is able to handle a maximum of 0.2% solids, the 6020A ICSA solution is higher in interfering components than any sample that would run through the instrument. However, the ICS solutions will be analyzed at levels that will provide approximately 0.1% dissolved solids.

12.8 Internal standards are used to correct for physical interferences. Masses used as internal standards include; ^{71}Ga, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh ^{45}Sc, and ^{89}Y. These internal standards are used in combination to cover the appropriate mass ranges. Internal standard correction is applied to the analytical isotopes via interpolation of the responses form nearest internal standard isotopes. This function is performed in real-time by the instruments operating system. Internal standards must be run within 50 AMU of the masses that are analyzed. Internal standard recoveries must fall between 30% and 125% when running method 6020, or 70% to 125% when running method 6020A Revision 1. If not, then the sample must be reanalyzed after a fivefold or greater dilution has been performed.

12.9 A method blank is digested and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or >½ the MRL for DoD projects or > 1/10 the sample result, corrective action must be taken. The MB can only be rerun once. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis.

12.10 Laboratory Control Samples are analyzed at a frequency of 5% or one per batch, whichever is greater. See the Attachments for a listing of control limits. For method 6020A, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. Project, QAPP, or client-specific control limits may supersede the limits listed, but laboratory limits should be consistent with specified limits in order to establish that the specified limits can be achieved. If the control limits are exceeded, the associated batch of samples will be redigested and reanalyzed.

12.11 A digested duplicate and matrix spike are analyzed at a frequency of 5% or one per batch, whichever is greater. The matrix spike recovery and relative percent difference will be calculated while analysis is in progress. See the Attachments for a listing of control limits. Project, QAPP, or client-specific control limits may supercede the limits listed. If the control limits are exceeded, the samples will be redigested and reanalyzed, unless matrix interference or sample non-homogeneity is established as cause. In these instances, the data and the report will be flagged accordingly.

12.12 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%). Default spike concentrations are listed in the sample digestion SOPs. Spike concentrations may be adjusted to meet project requirements. The matrix spike recovery will be calculated while the job is in progress. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria are derived from lab data and are listed in Table 2. For method

6020A, the recovery limits are 75-125%. If statistical in-house limits are used, they must fall within the 75-125% range. In some cases, project-specific QC limits may be required. Unless specified otherwise, for DoD QSM projects the project LCS criteria will be used for evaluation of matrix spikes. If an analyte recovery is outside acceptance limits proceed with the additional quality control tests described in sections 12.13 and 12.14. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. redigestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

Note: For DOD projects a MS/MSD is required with every extraction batch. The %RSD should be < 20%.

12.13 Post Digestion Spike Test: When analysis is conducted via 6020 a post digestion spike must be performed for each matrix and each batch of sample. The prepared sample or its dilution is spiked for each element of interest at a concentration sufficiently high to be observed. Typically 20 µL of 10,000 ppb intermediate stock is added to a 10 mL aliquot of sample. If analyte concentrations are elevated in the sample, spiking at a higher concentration may be required. The post spike should be recovered to within 75-125% of the known value or within the laboratory derived acceptance criteria. When analysis is conducted via 6020A, the post digestion spike test is performed whenever matrix spike or replicate criteria are exceeded. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. If this spike fails, then the dilution test (Sec. 12.14) should be run on this sample. If both the matrix spike and the post digestion spike fail, then matrix effects are confirmed.

12.14 Dilution Test: When analysis is conducted via 6020, a serial dilution test must be performed for each matrix and each batch of sample. For sample concentrations that are sufficiently high (minimally, a factor of greater than 100 times the MDL), the analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. When analysis is conducted via 6020A, the dilution test is performed whenever matrix spike or replicate criteria and post digestion spike criteria are exceeded. If the dilution test fails then a chemical or physical effect should be suspected. Corrective action can include additional dilution of the sample, the use of alternate methodologies, etc. or the data can be flagged and reported. The exact course of action will be dependent on the nature of the samples and project requirements and should be discussed with the project manager.

12.15 Instrument blanks should be evaluated for potential carryover and rinse times need to bring the analyte signal to within the CCB criteria discussed above in section 12.4. Results from instrument blanks run after standards or control samples should be used to establish levels at which carryover in samples may occur. Samples exhibiting similar effects of carryover should be reanalyzed.

12.16 Refer to the Quality Control section of EPA Methods 6020 and 6020A for additional information describing required QA/QC. Note that the nomenclature of certain QC samples in the method differs from that of the CLP SOW, but the function of those samples is equivalent in both cases.

13 DATA REDUCTION, REPORTING, AND REVIEW

13.1 Calculations

Calculate sample results using the data system printouts and digestion information. the digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = C^{*} \times Digestion\ Dilution\ Factor \times Post\ Digestion\ Dilution\ Factor$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

Solid samples are reported in mg/Kg:

$$mg/Kg(Sample) = C^{*} \times Post\ Digestion\ Dilution\ Factor \times \frac{Digestion\ Vol.(ml)}{Sample\ wt.(g)} \times \frac{1mg}{1000ug} \times \frac{1L}{1000ml} \times \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

NOTE: If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

13.2 Common isobaric interferences are corrected using equations equivalent to those listed in EPA Methods 6020, 6020A, and 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA methods for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3 Data Review and Reporting

13.3.1 The ICP-MS operator reviews the MS data and signs and dates the Data Review Form. A qualified senior staff spectroscopist performs a secondary review of the data and the Data Review Form is signed and dated. The data is then delivered to the report

generation area where it is filed in the service request file. Once all of the data for the service request is complete, a CAR is generated.

13.3.2 The data is saved on the local hard drive and is also copied to the appropriate directory on the network. The data directories are located at r:\icp\wip\data. The data is kept on the local directory for 1 month. The network files are periodically backed up on disc or network tape.

13.3.3 For "non-production" work (such as method development or research/development studies) the analyses are performed under the direction of a senior spectroscopist. All associated data is scrutinized by the senior spectroscopist. Original raw data and associated records are archived in the analytical project file.

13.3.4 The final review and approval of all data is performed by qualified spectroscopists.

14 CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

The method detection limit (MDL), limit of detection (LOD) and limit of quantitation (LOQ) are established using procedures described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL)*. Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16 TRAINING

16.1 A minimum of two senior level spectroscopists are to be maintained on staff at all times. Senior spectroscopists are defined as individuals with a minimum of ten years combined education and experience in, or related to atomic spectroscopy. Of those ten years, a minimum of two years of ICP-MS experience is required.

16.2 All technical staff is encouraged to attend one technical seminar per year. In addition to the technical seminars, senior spectroscopists are required to complete a one week training session offered by the instrument manufacturer.

16.3 Training outline

16.3.1 Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

16.3.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

16.3.3 Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

16.4 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

17 POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible and within method requirements. Standards are prepared in volumes consistent with the laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

18 WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

19 CHANGES SINCE THE LAST REVISION

19.1 Updated entire SOP to comply with EPA method 6020A requirements.

19.2 Sec 6.2 – Removed outdated information "Samples are generally received in the ICP-MS laboratory as 1-15% Nitric Acid digestates. Digestates originating from soil samples with greater than 60% solids are diluted prior to instrumental analysis by a factor of 5. This allows the analysis to achieve maximum sensitivity which results in optimum MRLs"

19.3 Sec 11.5.2 Added low level CRA acceptance criteria for 6020A.

19.4 The note at end of Sec 11.5.2 is new.

19.5 Sec 11.5.3, Added (MRL/LLICV/LLCCV) next to CRA.

19.6 Sec 12.2.1 and 12.2.2 Updated MDL SOP reference and requirements.

19.7 Sec 12.6.2, Added LOQ and removed NOTE from previous SOP from this section.

19.8 Sec 12.8, Added internal standard recovery criteria for method 6020A.

19.9 Sec 12.13 and 12.14 are new.

19.10 Remove Method EPA 200.8 from reference section

20 REFERENCES

20.1 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III Method 6020, Revision 0, September 1994.

20.2 USEPA, Test Methods for Evaluating Solid Waste, SW-846, Update IV, Method 6020A, Revision 1, February 2007.

20.3 VG and Thermo Elemental Instrument Manuals

TABLE 1
Method Reporting Limits and Method Detection Limits – Water Matrix

	Water (ug/L)			Water (ug/L)			Seawater (ug/L)		
	CLP Digestion			3020 Digestion			Reductive Precipitation		
Analyte	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**
Aluminum	2	**2**	**0.3**	2.4	**2**	**0.8**	-	-	-
Antimony	0.09	**0.05**	**0.03**	0.09	**0.05**	**0.03**	-	-	-
Arsenic	0.5	**0.5**	**0.08**	0.5	**0.5**	**0.07**	0.5	**0.5**	**0.02**
Barium	0.06	**0.05**	**0.02**	0.12	**0.05**	**0.04**			
Beryllium	0.02	**0.02**	**0.008**	0.02	**0.02**	**0.006**	0.02	**0.02**	**0.0007**
Bismuth	0.1	**0.1**	**0.02**	-	-	-	-	-	-
Boron	0.9	**0.5**	**0.3**	-	-	-	-	-	-
Cadmium	0.02	**0.02**	**0.008**	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.006**
Chromium	0.2	**0.2**	**0.07**	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Cobalt	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.001**
Copper	0.1	**0.1**	**0.02**	0.21	**0.1**	**0.07**	0.1	**0.1**	**0.03**
Lead	0.03	**0.02**	**0.009**	0.06	**0.05**	**0.02**	0.02	**0.02**	**0.003**
Manganese	0.06	**0.05**	**0.02**	0.05	**0.05**	**0.01**	-	-	-
Molybdenum	0.09	**0.05**	**0.03**	0.09	**0.05**	**0.03**	-	-	-
Nickel	0.2	**0.2**	**0.07**	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Selenium	1.2	**1**	**0.4**	1	**1**	**0.2**	-	-	-
Silver	0.03	**0.02**	**0.009**	0.03	**0.02**	**0.009**	0.02	**0.02**	**0.004**
Thallium	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.004**	0.02	**0.02**	**0.0009**
Tin	0.1	**0.1**	**0.04**	-	-	-	-	-	-
Uranium	0.02	**0.02**	**0.005**	0.02	**0.02**	**0.004**	-	-	-
Vanadium	0.2	**0.2**	**0.08**	0.2	**0.2**	**0.05**	-	-	-
Zinc	0.5	**0.5**	**0.1**	0.6	**0.5**	**0.2**	0.5	**0.5**	**0.04**

TABLE 1 (continued)
Method Reporting Limits and Method Detection Limits – Solid Matrix

	Soil/Sediment (mg/kg)			Tissue (mg/kg, dry basis)		
	3050 Digestion			PSEP		
Analyte	MRL (DoD)	**MRL**	**MDL**	MRL (DoD)	**MRL**	**MDL**
Aluminum	2	**2**	**0.5**	2	**2**	**0.4**
Antimony	0.09	**0.05**	**0.03**	0.06	**0.05**	**0.02**
Arsenic	0.5	**0.5**	**0.1**	0.5	**0.5**	**0.05**
Barium	0.09	**0.05**	**0.03**	0.15	**0.05**	**0.05**
Beryllium	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.005**
Bismuth	0.1	**0.1**	**0.02**	-	-	-
Cadmium	0.02	**0.02**	**0.008**	0.03	**0.02**	**0.01**
Chromium	0.2	**0.2**	**0.04**	-	-	-
Cobalt	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.006**
Copper	0.3	**0.1**	**0.1**	0.1	**0.1**	**0.03**
Lead	0.06	**0.05**	**0.02**	0.02	**0.02**	**0.006**
Manganese	0.12	**0.05**	**0.04**	0.06	**0.05**	**0.02**
Molybdenum	0.15	**0.05**	**0.05**	0.06	**0.05**	**0.02**
Nickel	0.2	**0.2**	**0.05**	0.2	**0.2**	**0.03**
Selenium	2	**1**	**0.4**	-	-	-
Silver	0.06	**0.02**	**0.02**	0.02	**0.02**	**0.006**
Thallium	0.02	**0.02**	**0.003**	0.02	**0.02**	**0.005**
Tin	0.2	**0.1**	**0.06**	-	-	-
Uranium	0.02	**0.02**	**0.004**	0.02	**0.02**	**0.007**
Vanadium	0.2	**0.2**	**0.04**	0.2	**0.2**	**0.04**
Zinc	0.6	**0.5**	**0.2**	1.2	**0.5**	**0.4**

ATTACHMENTS

List of Target Element Masses

Example Standard Sheet

QC Acceptance Criteria

Analyte	ISOTOPES ANALYZED	ISOTOPE REPORTED
Aluminum	27	27
Antimony	121,123	123
Arsenic	75	75
Barium	135,137,138	137
Beryllium	9	9
Cadmium	111,112,114	111
Chromium	52,53	52
Cobalt	59	59
Copper	63,65	65
Lead	206,207,208	208
Manganese	55	55
Molybdenum	95,97,98	98
Nickel	60,61,62	60
Selenium	77,78,82	82
Silver	107,109	107
Thallium	203,205	205
Uranium	238	238
Vanadium	51	51
Zinc	66,67,68	66

METALS SPIKE FORM

Service Request #_____
Q.C. Sample #_____

Circle type of digest: GFAA ICP FAA ICP-MS Other:_____ Initials / Date:_____/_____
Circle type of sample: Soil Water Misc. Sludge Oil Other:_____

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Enter mls Added	
K-MET SS1 ID#14929	HNO3	50.0	1000ml	-		Expires:05/01/2010
	Al	100*	1000ml	200		
	Ag	100*	1000ml	5		
	Ba	100*	1000ml	200		
	Be	100*	1000ml	5		
	Cd	100*	1000ml	5		
	Co	100*	1000ml	50		
	Cr	100*	1000ml	20		
	Cu	100*	1000ml	25		
	Fe	100*	1000ml	100		
	Pb	100*	1000ml	50		
	Mn	100*	1000ml	50		
	Ni	100*	1000ml	50		
	Sb	50	1000ml	50		
	V	100*	1000ml	50		
	Zn	100*	1000ml	50		
K-MET SS2 ID#11468	HNO3	25.0	500ml	-		Expires:4/2010
	As	2.0	500ml	4		
	Cd	2.0	500ml	4		
	Pb	2.0	500ml	4		
	Se	2.0	500ml	4		
	Tl	2.0	500ml	4		
	Cu	2.0	500ml	4		
K-MET SS3 ID#15279	HNO3	25.0	500ml	-		Expires:05/2010
	As	50.0	500ml	100		
	Se	50.0	500ml	100		
	Tl	50.0	500ml	100		
K-MET SS4 ID#14962	HNO3	25	500ml	-		Expires:7/30/2010
	B	50	500ml	100		
	Mo	50	500ml	100		
K-MET SS5 ID#15720	HNO3	10.0	200ml	-		Expires 3/29/10
	K**	20	200ml	1000		
	Na**	20	200ml	1000		
	Mg**	20	200ml	1000		
	Ca**	20	200ml	1000		
K-MET GFLCSW ID# 15022	HNO3	10.0	1000ml	-		Expires: 07/01/10
	As, Pb, Se, Tl	5.0	1000ml	2.5		
	Cd	-	-	1.25		
	Cu	2.5	1000ml	2.5		
K-MET QCP-CICV-1 ID# 12779	Ca, Mg, Na, K	no dilution	-	2500		Expires:10/01/10
	Al, Ba	no dilution	-	1000		
	Fe	no dilution	-	500		
	Co, Mn, Ni, V, Zn	no dilution	-	250		
	Cu, Ag	no dilution	-	125		
	Cr	no dilution	-	100		
	Be	no dilution	-	25		
K-MET QCP-CICV-2 ID# 12778	Sb	no dilution	-	500		Expires: 12/10
K-MET QCP-CICV-3 ID#14972	As, Pb, Se, Tl	no dilution	-	500		Expires:12/1/10
	Cd	no dilution	-	250		

* Denotes volume of mixed stock standard.
** Denotes 10,000 ppm individual stock standards.

Standard	mls of standard	ppm	Logbook #	Exp. Date

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
6020	3050B	Soil	Aluminum	41-158	75-125*	20
6020	3050B	Soil	Antimony	50-150	10-103	20
6020	3050B	Soil	Arsenic	78-122	57-133	20
6020	3050B	Soil	Barium	81-119	54-173	20
6020	3050B	Soil	Beryllium	83-117	64-133	20
6020	3050B	Soil	Boron	67-133	75-125*	20
6020	3050B	Soil	Cadmium	81-119	68-137	20
6020	3050B	Soil	Chromium	80-119	34-175	20
6020	3050B	Soil	Cobalt	82-118	74-118	20
6020	3050B	Soil	Copper	83-116	22-181	20
6020	3050B	Soil	Lead	79-121	27-178	20
6020	3050B	Soil	Manganese	81-119	75-125*	20
6020	3050B	Soil	Molybdenum	75-125	53-143	20
6020	3050B	Soil	Nickel	81-118	59-132	20
6020	3050B	Soil	Selenium	80-120	65-125	20
6020	3050B	Soil	Silver	66-134	62-131	20
6020	3050B	Soil	Thallium	79-120	70-128	20
6020	3050B	Soil	Uranium	80-120*	75-125*	20
6020	3050B	Soil	Vanadium	79-121	59-142	20
6020	3050B	Soil	Zinc	73-121	37-162	20
6020	CLP/3020A	Water	Aluminum	85-120	56-143	20
6020	CLP/3020A	Water	Antimony	91-112	66-133	20
6020	CLP/3020A	Water	Arsenic	89-112	72-129	20
6020	CLP/3020A	Water	Barium	92-111	86-117	20
6020	CLP/3020A	Water	Beryllium	81-122	73-125	20
6020	CLP/3020A	Water	Cadmium	92-111	87-113	20
6020	CLP/3020A	Water	Chromium	88-113	60-136	20
6020	CLP/3020A	Water	Cobalt	87-114	84-115	20
6020	CLP/3020A	Water	Copper	89-113	62-130	20
6020	CLP/3020A	Water	Lead	90-112	76-117	20
6020	CLP/3020A	Water	Manganese	89-115	25-180	20
6020	CLP/3020A	Water	Molybdenum	66-135	67-138	20
6020	CLP/3020A	Water	Nickel	89-113	78-117	20
6020	CLP/3020A	Water	Selenium	87-115	47-150	20
6020	CLP/3020A	Water	Silver	64-134	55-136	20
6020	CLP/3020A	Water	Thallium	78-123	75-121	20
6020	CLP/3020A	Water	Vanadium	87-113	82-119	20
6020	CLP/3020A	Water	Zinc	86-119	65-126	20

STANDARD OPERATING PROCEDURE

for

MERCURY IN SOLID OR SEMISOLID WASTE

SOP No.: MET-7471A

Revision: 14

July 28, 2009

Approved by: _____ Supervisor — 7/30/09 Date

_____ QA Manager — 7/30/9 Date

_____ Laboratory Manager — 7/30/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

MERCURY IN SOLID OR SEMISOLID WASTE

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Mercury in soils, sediments, freeze dried tissues, bottom deposits, and sludge-type materials. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix. Method 7471A is a cold-vapor atomic absorption procedure.

1.2. The Method Reporting Limit (MRL) is 0.02 mg/kg. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. A Method Detection Limit (MDL) of 0.006 mg/kg has been achieved using this procedure.

2. METHOD SUMMARY

A representative aliquot of sample is prepared as described in this procedure. The mercury is reduced to its elemental state and aerated from solution and measured with an atomic absorption spectrometer. The mercury vapor passes through a cell positioned in the light path of the AA where absorbance is measured as a function of mercury concentration.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2. **Independent Calibration Verification** (ICV) - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3. **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.4. **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample

digestion and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.5. **Duplicate Sample** (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.6. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.7. **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8. **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis

4. INTERFERENCES

Potassium permanganate is added to eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which absorbs radiation at 253 nm.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE PRESERVATION AND STORAGE

6.1. Non-aqueous samples are stored at 4 ± 2 °C from receipt until analysis, unless otherwise dictated by project specifications.

7. APPARATUS AND EQUIPMENT

7.1. CETAC M-6000A Mercury Analyzer. See Attachments for instrument parameters.

7.2. CPI-Modified Block (Mod Block)

7.3. Pipettors, Eppendorf and Finnpipette fixed and adjustable volume

7.4. Polypropylene graduated cylinders, 25 mL

7.5. 125 ml Digestion Vessel tubes.

7.6. Laboratory balance, top-loader capable of readings .001g (3-place). Mettler, Ohaus, or equivalent.

8. STANDARDS AND REAGENTS

8.1. Mercury stock solution (1,000 mg/L). Commercially prepared certified solution stored at room temperature. The expiration date determined by manufacturer.

8.2. Mercury working standard (100µg/L). Prepared from the intermediate stock solution listed above. Store at room temperature and prepare a new standard daily.

8.3. Laboratory Control Sample – ERA Priority Pollutant/CLP Inorganic Soil reference material. Store at room temperature in the original container and use the vendor expiration date.

8.4. Matrix spike solution (1 mg/L) – Prepare by making a 1:1000 dilution of the mercury stock solution. Store at room temperature and prepare a new standard monthly.

Note: See section 11.2.2 for details on preparation of calibration and ICV standards. See section 12 for QC sample preparation.

8.5. Reagent water - ASTM Type II water (laboratory deionized water).

8.6. Acids - Purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the Method Reporting Limit.

8.6.1. Nitric Acid (HNO_3) 69-70% – JT Baker-Baker Instra-Analyzed® or equivalent.
8.6.2. Sulfuric Acid concentrated (H_2SO_4) – EMD-OmniTrace® or equivalent.
8.6.3. Hydrochloric Acid concentrated (HCL) – VWR – BHD-Aristar® or equivalent.

8.7. Potassium permanganate solution, 5% w/v. To prepare, add 50 g of solid reagent to 1000 mL of D.I. water and place on magnetic stir plate for a approximately 30 minutes until dissolved.

8.8. Sodium chloride/hydroxylamine hydrochloride solution, 12% w/v each. To prepare, add 120g sodium chloride and 120 g of hydroxylamine hydrochloride to 1000 mL of D.I. water and place on magnetic stir plate for a approximately 15 minutes until dissolved.

8.9. Stannous chloride, 10% w/v in HCl (7% v/v). To prepare, add 100g stannous chloride crystals and 70 mL of concentrated hydrochloric acid in 1000 mL of D.I. water. Seal lid on mixing bottle and shake until the stannous chloride is dissolved.

8.10. Aqua Regia – Prepare immediately before use by carefully adding 3 parts of concentrated HCL to one part of HNO_3.

9. PREVENTIVE MAINTENANCE

9.1. CAS staff performs all routine maintenance and troubleshooting. Preventative maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Repairs of an extraordinary nature may or may not require factory service, depending on the nature of the task. All maintenance activities are recorded in a maintenance logbook.

9.2. Keep the instrument free of dust, deposits, and chemical spills.

9.3. Replace the peristaltic and autosampler rinse tubing.

9.4. Remove and clean the Gas-Liquid Separator.

9.5. Remove, dismantle, and clean the optical cells (sample cell and reference cell) including the sapphire windows.

9.6. Replace the Hg lamp bulb when the lamp current reaches 13 mA.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for *Documentation of Training*, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Mix the sample thoroughly to achieve homogeneity. For soil, sediment, solids, weigh approximately 0.5g of well-homogenized sample and place in the bottom of a 125 ml digestion tube and record the weight to the nearest 0.01g. Add 5.0 mL of reagent water and 5.0 mL of aqua regia, then heat in the Mod Block for 2 minutes at 95°C.

11.1.2. Cool then add 10 mL of reagent water and 15 mL of potassium permanganate solution. If the purple color does not persist for 15 minutes add additional potassium permanganate until it does so. Any additional potassium permanganate solution must also be added to the blanks and standards in equal proportion. **Note:** Spiking solution is added prior to acidification.

11.1.3. Mix thoroughly and place in the heating block for 30 minutes at 95°C. The temperature of the block is monitored with a thermometer that is calibrated monthly.

11.1.4. Cool and add 6 mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate. Perform this addition under a hood as Cl_2 could be evolved.

11.1.5. Add 27 mL of reagent water and the sample is ready for analysis. (The vapor generator does the step of adding the stannous chloride solution automatically.)

11.2. Calibration

11.2.1. To prepare calibration standards a 10 ppm intermediate stock solution is first prepared by aliquoting 1.0 mL of commercially prepared 1000 ppm stock standard into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared monthly. Next, a 100 ppb working solution is prepared by aliquoting 1.0 mL of the 10 ppm intermediate stock solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared daily.

Note: All standard aliquots are measured using calibrated fixed or adjustable volume autopipetters or calibrated disposable 5.0 or 10.0 mL pipettes.

11.2.2. Transfer 0, 0.1, 0.25, 0.5, 2.5 and 5.0 mL aliquots of the working solution to a series of labeled 125 ml digestion tubes. Add the appropriate amount of reagent water to bring each bottle to a volume of 5mL. Add 5.0 mL of aqua regia and heat in the heating block for 2 minutes at 95°C. The final concentrations of the prepared standards are 0, 0.2, 0.5, 1.0, 5.0, 10.0 ppb.

The Initial Calibration Verification (ICV) is prepared by first making a 1000 ppb intermediate solution. 0.10 mL of commercially prepared 1000 ppm stock standard, from a different manufacturer and lot than the calibration standard, is aliquoted into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This solution must be prepared monthly. Prepare the ICV standard by aliquoting 0.25 mL to a labeled 125 ml digestion tube. Add the 4.75 mL of reagent water and 5.0 ml of Aqua Regia.

11.2.3. Cool and then add 10 mL of reagent water and 15 mL of potassium permanganate solution and return the bottles to the water bath for 30 minutes.

11.2.4. Cool and add 6.0 mL of sodium chloride-hydroxylamine hydrochloride solution. Add 27 mL of reagent water and the standards are ready for analysis.

11.2.5. CETAC Calibration and Sample Analysis

11.2.5.1. Turn on the CETAC instrument, including the Hg lamp, and autosampler. After this is done turn open the operating software (Mercury Analyzer 1.5.1.1).

11.2.5.2. The rinse station for the autosampler turns on automatically, but the peristaltic pump must be started manually. Make sure all sample uptake and drain tubes are placed correctly on the pump and are secured with the appropriate tension. Place the reagent uptake tube in the stannous chloride and start the pump.

11.2.5.3. From the software's main screen select the "Worksheet" button and then the "Template" button. Select the "Kelso Mercury Program".

11.2.5.4. Go to the "Labels" tab and enter the QC and field samples to be analyzed in the appropriate order.

11.2.5.5. Transfer the solutions to be analyzed to labeled 12mL polyethylene test tubes and place them in the appropriate spaces on the autosampler trays.

11.2.5.6. Transfer the calibration blank and standards (0.2, 0.5, 1.0, 5.0, and 10 ppb) from their digestion tubes to the standard tubes located behind the autosampler trays. The calibration blank is placed in the left most tube and the other standards are placed in ascending order to the right.

11.2.5.7. Return to the software and go to the "Analysis" tab. At this point the analysis is ready to begin. Click on the start button. In the dialog box that appears be sure the following are checked:

- Calibrate before first sample.
- New output file before first sample.
- Zero before first sample.

Click start and the analysis will begin.

11.2.6. After the calibration standards have run the software will use linear regression to create a calibration curve based on the concentration and measured absorbance of each standard. The form of regression line is $y = mx + b$. If the correlation coefficient of the curve is greater than 0.995 the analysis will continue, if not the analysis will be terminated and corrective action will be needed by the analyst.

11.3. As the analysis sequence proceeds, next analyze the following QC standards.

- ICV (5.0 ppb standard prepared from a second source)
- ICB
- CCV (5.0 ppb calibration standard)
- CCB
- CRA (0.2 ppb calibration standard)

If either the ICV or CCV are different from their true values by more than 10% the software will terminate the analysis. If either the ICB or CCB is greater than the MRL the software will terminate the analysis. Method 7471A does not contain criteria for the CRA, however, the result must be a positive measured concentration. For 7471B analyses the criteria are 70-130% of the true value. Also, specific project requirements may apply.

Note: For projects falling under DoD QSM requirements, the QSM criteria for CCV standards is ±20% and for ICB and CCB standards no analytes detected > LOD. (The ICV limit is as listed above.)

11.4. Sample Analysis

11.4.1. The samples are analyzed with the CETAC analyzer in the same manner as the calibration standards. The analyzer does the step of adding the stannous chloride solution automatically. Check the baseline between samples to verify that the spectrometer reading has stabilized at the normal baseline level.

11.4.2. The analytical sequence should be set up to include all samples, QC samples, blanks, and calibration verification standards at necessary intervals. Refer to the SOP for Sample Batches.

11.4.3. Sample digestion batches are analyzed with a set of CCV and CCB standards which are run at the beginning and end of the analytical run and at a minimum every 10 samples during the run. The same criteria listed above are applied to the CCVs and CCBs and if one is found to be outside these limits the analysis is terminated.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. Acceptable accuracy and precision of the procedure must be demonstrated before analysis of samples begins, or whenever significant changes to the procedures have been made.

12.1.2. Accuracy and precision is demonstrated by preparing and analyzing four LCS aliquots. The average percent recovery of for each analyte must be within LCS limits and the %RSD within precision limits.

12.1.3. Initial demonstration of capability must be performed by each analyst performing sample analysis and documented in the laboratory records.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution near the MRL and analyze. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL).

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually. The MDL study and MDL verification check should be analyzed annually or whenever there are major changes in the instrument or procedure is implemented.

12.3. For method 7471B, an LLQC sample (a CRA that is carried through the digestion) must be analyzed to verify accuracy at the MRL. The recovery must be 70-130%.

12.4. For method 7471B, Instrument Detection Limit (IDL) studies are performed quarterly. These will be calculated and made available to the analysts.

12.5. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual, in the *SOP for Sample Batches* (ADM-Batch). For this analysis, these include:

12.5.1. Prepare one method blank (MB) per digestion batch, or per 20 samples, whichever is more frequent. The MB is to be prepared as done with samples. The Method Blank should be < MRL. If the Method Blank is >MRL redigest the associated samples if sample levels are <20x the MB level.

Note: For projects falling under DoD QSM requirements, the QSM criteria for method blanks is no analytes detected > ½ MRL.

12.5.2. Prepare one Laboratory Control Sample (LCS) per digestion batch, or per 20 samples. Weigh 0.25g of the current lot of "Environmental Resource Associates PriorityPollutnT/CLP Inorganic Soil" prepared reference material in to a 125 mL Digestion vessel tube and prepare as per the procedure.

The LCS recovery criteria are 72-122%, unless project-specific or in-house limits are established. For method 7471B, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range.

Note: For DoD QSM projects, the QSM LCS criterion is 80-120%. If the LCS fails the acceptance criteria, redigest the batch of samples.

12.5.3. Prepare one sample duplicate and one matrix spike sample per each digestion batch, or per twenty samples, whichever is more frequent. For the matrix spike, add 0.25mL of the matrix spike solution to the designated spike sample, resulting in a spike concentration of 0.5 mg/kg. At times, specific samples will be assigned as duplicates or spikes depending on client requirements.

Note: Duplicate samples are routinely analyzed; however some projects may require a MSD. All DoD projects require a MSD. The MSD sample is prepare as described above.

The RPD criterion for duplicates is 30% RPD. If not, flag the data or redigest samples. A matrix spike recovery criterion is 60-135%, unless project-specific limits are required. For method 7471B, the recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. For DoD QSM work, MS recoveries are assessed using the QSM LCS control limits. If the MS (and/or MSD where applicable) recovery is outside acceptance limits proceed with the additional interference tests described in section 12.5.4. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. redigestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required.

Note: For DoD QSM projects, the duplicate RPD limit is 20% and MS recoveries are assessed using the QSM LCS control limits 80-120%.

12.5.4. Interference Tests: Prepare one post spike for every batch of samples and if samples are sufficiently high (10x the MRL/LOQ) a serial dilution. The serial dilution must agree within 10% of the original sample result. Post spike recovery acceptance limits for method 7470A are 85-115% under SW846 Update III, and 80-120% for project falling under SW846 Update IV. When both the post spike and dilution tests fail all of the samples in the associated preparation batch must be quantified via Method of Standard Additions (MSA).

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12.

13.2. Record all sample weight, volumes and dilutions on an A.A. benchsheet (see Attachments).

13.3. Solution concentrations are calculated by the Mercury Analyzer software based on the linear regression calibration curve created when the calibration standards are analyzed. The absorbance measured for each sample is applied to the linear regression curve and the final solution concentration is determined and displayed as the instrument result.

13.4. Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result. Solid samples are reported in mg/Kg:

$$mg/Kg(Sample) = C^{*} \times PostDigestionDilutionFactor \times \frac{DigestionVol.(ml)}{Samplewt.(g)} \times \frac{1mg}{1000ug} \times \frac{1L}{1000ml} \times \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

NOTE: If results are to be reported on a dry weight basis as required by certain projects, the Sample Wt(g) component of the equation should be the dry-weight derived from a determination of %moisture of a separate aliquot of the sample using the SOP for Total Solids.

13.5. Record all concentrations determined at the instrument and calculate the final results in mg/Kg. Record the final results on the A.A. Benchsheet.

13.6. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\ICP\WIP. Once the results are transferred, the report is reviewed.

13.7. A daily run log of all samples analyzed is maintained. All data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.8. Refer to the SOP for Laboratory Data Review Process for general instructions for data review.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.2. The method detection limit (MDL), limit of detection (LOD), and limit of quantitation (LOQ) are established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP *for Documentation of Training* (ADM-TRANDOC).

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Final Update II, Method 7471A, September 1994.

19.2. USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update IV, Method 7471B, Revision 2, February 2007.

19.3. DoD Quality Systems Manual for Environmental Laboratories Version 4.1 4/22/2009.

20. CHANGES SINCE THE LAST REVISION

20.1. Sec 8.10 Added procedure for preparing aqua regia.
20.2. Sec 11 Changed ratio of reagents to sample to conform to EPA method/
20.3. Sec 11.3 Added DoD CCV limits.
20.4. Sec 12.5.3 Added DoD MSD requirement and clarification language on assessing MSD. Added DoD RPD criteria.
20.5. Sec 12.5.4 Added interference check procedure and acceptance criteria.
20.6. Sec 15.2 Added LOD.
20.7. Sec 19 Added DoD QSM reference

ATTACHMENTS

Instrument Parameters
Benchsheets

Analyst	M SMITH
Date Created:	Tuesday, August 04, 2009
Worksheet	Hg 080409B
Comment	K-CVAA-01
Sip Duration (Sec.):	40
Rinse Duration (Sec.):	60
Read Delay:	45
Integration Time/Replicate:	3.00
# of Replicates:	4
# of Repeats:	1
Baseline Correction Enabled:	True
Baseline Point 1 Start Time:	7
Baseline Point 1 End Time:	10
2-Point Baseline Corr. Enabled:	False
Baseline Point 2 Start Time:	
Baseline Point 2 End Time:	
Gas Flow (ml/min):	135
Calibration Algorithm:	Linear, Zero Intercept
Recalibration Frequency:	0
Reslope Frequency:	0
Reslope Standard:	4
Calibration Standard #1 Conc.:	0.20 PPB
Calibration Standard #2 Conc.:	0.50 PPB
Calibration Standard #3 Conc.:	1.00 PPB
Calibration Standard #4 Conc.:	5.00 PPB
Calibration Standard #5 Conc.:	10.00 PPB
QC Enabled:	True
QC-RSD Enabled:	False
QC-Std Enabled:	False
QC-Blank Enabled:	False

CVAA Mercury Data Review Form

Element: Hg

Analysis Lot #: _____

Cal. STD/CCV Source: _____

Service Request Numbers:

_____

_____

_____

	Yes	No	NA
1) Appropriate standardization completed	_____	_____	_____
2) ICV within 10% of true value	_____	_____	_____
3) CCVs in control	_____	_____	_____
4) CCBs and or ICBs below MRL	_____	_____	_____
5) All reported samples within calibration range	_____	_____	_____
6) Calculations correct	_____	_____	_____

Comments:

Data reviewed against service request(s) to ensure no samples were omitted: _____ (initials)

Primary Reviewed By: _____ Date: _____

Secondary Reviewed By: _____ Date: _____

COLUMBIA ANALYTICAL SERVICES, INC. PRINTOUT WITH:_____
ANALYTICAL WORKSHEET

Method: (Circle One) 7470A 7471A 245.1	Service Request # :
Analysis For: Hg	

DATA

Pos.	SAMPLE NUMBER	Initial Sample (g) or (mL)	Initial Dilution (mL)	Dilution Factor	(µg/L) Measured	Sample Actual (mg/kg)	Sample Actual (µg/L)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							

Comments: Reporting Levels:

Soil/Tissue Spike Level:

Post Spike Level: x @ 5 µg/L

Method	Spike Level	MRL	LCS Limit	MS Limit	RPD
7470A Water	1.0 µg/L	0.2 µg/L	84-117%	78-122%	20%
245.1 Water	1.0 µg/L	0.2 µg/L	85-115%	70-131%	20%
7470A TCLP	5.0 µg/L	1.0 µg/L	85-115%	75-125%	20%
7471A Soil LCSS	1.77 mg/kg	0.02 mg/kg	76-121%	64-127%	30%
7471A Tissue Tort	0.27 mg/kg	0.02 mg/kg	78-122%	60-130%	30%

Analyst:	Date:	Page Number: 1

COLUMBIA ANALYTICAL SERVICES, INC. PRINTOUT WITH:_____
ANALYTICAL WORKSHEET

Method: (Circle One) 7470A 7471A 245.1	Service Request # :
Analysis For: Hg	

DATA

Pos.	SAMPLE NUMBER	Initial Sample (g) or (mL)	Initial Dilution (mL)	Dilution Factor	Measured (µg/L)	Measured Adjusted (µg/L)	Sample Actual (mg/kg)	Sample Actual (µg/L)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Comments: Reporting Levels:

Soil/Tissue Spike Level:

Post Spike Level: x @ 5 µg/L

Method	Spike Level	MRL	LCS Limit	MS Limit	RPD
7470A Water	1.0 µg/L	0.2 µg/L	84-117%	78-122%	20%
245.1 Water	1.0 µg/L	0.2 µg/L	85-115%	70-131%	20%
7470A TCLP	5.0 µg/L	1.0 µg/L	85-115%	75-125%	20%
7471A Soil LCSS	1.77 mg/kg	0.02 mg/kg	76-121%	64-127%	30%
7471A Tissue Tort	0.27 mg/kg	0.02 mg/kg	63-148%	60-130%	30%

Analyst:	Date:	Page Number: 1

Columbia Analytical Services

EPA METHOD 7470A

Service Request Number(s) :
PREP RUN:

Sample	Initial Volume	Final Volume	Sample	Initial Volume	Final Volume
MB	20	20			
LCSW					
Std. 0.2	0.2 *				100
Std. 0.5	0.5 *				100
Std. 1.0	1.0 *				100
Std. 5.0	5.0 *				100
Std. 10.0	10.0 *				100
ICV	0.5 **				100

Start Time: Finish Time: Waterbath Temp.: 95° C

Balance#: 1

Lot # of Reagents Used:		
HNO_3: G20046	$K_2S_2O_8$: C45H07	NaCl : E29582
H_2SO_4: 46137	$KMnO_4$: G19476	NH_2OH-HCL: E31591
HCL: 10286	$SnCl_2$: G20637	ERA CLP Soil: D045540

* Source Standard: _____ 100 ppb Spike = ml * Source Standard

**Source Standard: ICV 1000 ppb LCSW= ml ICV **Source Standard

Comments:	
Analyst:	Date:

Columbia Analytical Services

EPA METHOD 7470A

Service Request Number(s) :
PREP RUN:

Sample	Initial Volume	Final Volume	Sample	Initial Volume	Final Volume
MB	20	20			
LCSW					
Std. 0.2	0.2 *				100
Std. 0.5	0.5 *				100
Std. 1.0	1.0 *				100
Std. 5.0	5.0 *				100
Std. 10.0	10.0 *				100
ICV	0.5 **				100

Start Time: Finish Time: Waterbath Temp.: 95° C

Lot # of Reagents Used:		
HNO_3: G20046	$K_2S_2O_8$: C45H07	NaCl : E29582
H_2SO_4: 46137	$KMnO_4$: G19476	NH_2OH-HCL: E31591
HCL: 10286	$SnCl_2$: G20637	ERA CLP Soil: D045540

* Source Standard: _____ 100 ppb

**Source Standard: ICV _____ 1000 ppb Spike/LCSE = _____ ml _____ 1000 ppb

Comments:	
Analyst:	Date:

Columbia Analytical Services

EPA METHOD 245.1

Service Request Number(s) :
PREP RUN:

Sample	Initial Volume	Final Volume	Sample	Initial Volume	Final Volume
MB	100	100			
LCSW					
Std. 0.2	0.2 *				100
Std. 0.5	0.5 *				100
Std. 1.0	1.0 *				100
Std. 5.0	5.0 *				100
Std. 10.0	10.0 *				100
ICV	0.5 **				100

Start Time: **Finish Time:** **Waterbath Temp.:** 95° C

Lot # of Reagents Used:		
HNO_3: G20046	$K_2S_2O_8$: C45H07	NaCl : E29582
H_2SO_4: 46137	$KMnO_4$: G19476	NH_2OH-HCL: E31591
HCL: 10286	$SnCl_2$: G20637	ERA CLP Soil: D045540

* Source Standard: _____ 100 ppb Spike = ml * Source Standard

**Source Standard: ICV_____ 1000 ppb LCSW= ml ICV **Source Standard

Comments:	
Analyst:	Date:

Columbia Analytical Services

EPA METHOD 7471A

Service Request Number(s) :
PREP RUN:

Sample	Wet Weight (g)	% Solids	Dry Weight (g)	Final Volume (mL)
MB	~	~		50
LCS	~	~		
Std. 0.2	0.1 * mL			50
Std. 0.5	0.25 * mL			50
Std. 1.0	0.5 * mL			50
Std. 5.0	2.5 * mL			50
Std. 10.0	5.0 * mL			50
ICV	0.25 ** mL			50

Start Time: Finish Time: Hot block Temp.: 95° C

Balance#: 1

Lot # of Reagents Used:		
HNO_3: G20046	$K_2S_2O_8$: C45H07	NaCl : E29582
H_2SO_4: 46137	$KMnO_4$: G19476	NH_2OH-HCL: E31591
HCL: 10286	$SnCl_2$: G20637	ERA CLP Soil: D045540

* Source Standard: _____ 100 ppb Spike = ml _____ 1000 ppb

**Source Standard: ICV _____ 1000 ppb LCSW= ml ICV **Source Standard

Comments:	
Analyst:	Date:

Columbia Analytical Services

EPA METHOD 7471A (Tissue)

Service Request Number(s) :
PREP RUN:

Sample	Wet Weight (g)	% Solids	Dry Weight (g)	Final Volume (mL)
MB	~	~		50
LCS Tort		92.6		
	~	~		
	~	~		
	~	~		
	~	~		
	~	~		
	~	~		
	~	~		
	~	~		
Std. 0.2	0.1 * mL			50
Std. 0.5	0.25 * mL			50
Std. 1.0	0.5 * mL			50
Std. 5.0	2.5 * mL			50
Std. 10.0	5.0 * mL			50
ICV	0.25 ** mL			50

Start Time: **Finish Time:** **Hot plate/Waterbath Temp.: 95° C**

Balance#: 1

Lot # of Reagents Used:		
HNO_3: G20046	**$K_2S_2O_8$: C45H07**	**NaCl : E29582**
H_2SO_4: 46137	**$KMnO_4$: G19476**	**NH_2OH-HCL: E31591**
HCL: 10286	**$SnCl_2$: G20637**	**ERA CLP Soil: D045540**

* Source Standard: _____ 100 ppb Spike = ml _____ 1000 ppb

**Source Standard: ICV_____ 1000 ppb LCSW= ml ICV **Source Standard

Comments:	
Analyst:	**Date:**

Columbia Analytical Services

EPA METHOD 7470A

Service Request Number(s) :
PREP RUN:

Sample	Initial Volume	Final Volume	Sample	Initial Volume	Final Volume
MB	20	20			
LCSW					
Std. 0.2	0.2 *				100
Std. 0.5	0.5 *				100
Std. 1.0	1.0 *				100
Std. 5.0	5.0 *				100
Std. 10.0	10.0 *				100
ICV	0.5 **				100

Start Time: Finish Time: Waterbath Temp.: 95° C

Balance#: 1

Lot # of Reagents Used:		
HNO_3: G20046	$K_2S_2O_8$: C45H07	NaCl : E29582
H_2SO_4: 46137	$KMnO_4$: G19476	NH_2OH-HCL: E31591
HCL: 10286	$SnCl_2$: G20637	ERA CLP Soil: D045540

* Source Standard: _____ 100 ppb Spike = ml * Source Standard

**Source Standard: ICV_____ 1000 ppb LCSW= ml ICV **Source Standard

Comments:	
Analyst:	Date:

STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

SOP No.: MET-ICP

Revision: 22
July 30, 2010

Approved by: _____ Supervisor — 7/13/10 Date

_____ QA Manager — 7/15/10 Date

_____ Laboratory Manager — 7/15/10 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

1 SCOPE AND APPLICATION

1.1 This procedure describes the steps taken for the analysis of soil, sludge surface water and drinking water digestates using EPA methods 6010C, 200.7, and CLP ILM04.0 for a variety of elements. The digested samples and QC standards are all diluted in a similar acid matrix. A procedure is also given for calculation of hardness by Standard Methods 2340B.

1.2 The Method Reporting Limits (MRLs) for common elements are listed in Table 1. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are listed in Table 1. The MDL and MRL may change as annual studies are performed.

1.3 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP or project which require older versions of EPA methods (i.e. 6010B). QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2 METHOD SUMMARY

2.1 A representative aliquot of sample is prepared as described in the applicable digestion SOP. The digestate is analyzed for the elements of interest using ICP spectrometry. The instrument measures characteristic emission spectra by optical spectrometry. The intensity of emission lines are monitored.

2.2 Final results are calculated using the digestion information and the results from the ICP analysis. Data is reported using standard CAS procedures and formats, or following project specific reporting specifications.

2.3 Deviations from the reference method(s): This SOP contains no deviations from the reference methods.

3 DEFINITIONS

3.1 **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2 **Independent Calibration Verification** (ICV) - ICV solutions are made from stock solutions different from the stock used to prepare calibration standards and are used to verify the validity of the standardization.

3.3 **Laboratory Control Sample** (LCS): A laboratory blank that has been fortified with target analyte and used to determine that the analysis is in control. For solids, a reference material may be used unless prohibited by project protocols.

3.4 **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.5 **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.6 **Duplicate Sample** (DUP) - A laboratory duplicate is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.7 **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.8 **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.9 **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

3.10 **Laboratory fortified Blank (LFB)**- A laboratory blank that has been fortified with target analyte at the method reporting limit and used to determine if the laboratory can detect contaminants at the method reporting limit.

4 INTERFERENCES

4.1 Interferences from contaminated reagents must be eliminated. The purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the MRL (or above ½ the RL for DoD work).

4.2 Background emission and stray light can be compensated by background correction.

4.3 Spectral overlaps resulting in interelement contributions can be corrected for by using interelement correction factors. Interelement correction factors are established for each instrument and are maintained by the analyst at the workstation.

5 SAFETY

5.1 Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.2 Hydrochloric, Nitric and Hydrofluoric Acids are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. Safety glasses, lab coat and gloves should be worn while working with the solutions.

5.3 High Voltage - The power unit supplies high voltage to the RF generator which is used to form the plasma. The unit should never be opened. Exposure to high voltage can cause injury or death.

5.4 UV Light -The plasma when lit is a very intense light, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Samples are prepared using methods 3005A, 3010A, 3050, or CLPILM04.0 (CAS SOPs MET-3005A, MET-3010A, MET-3050, and MET-DIG). Samples are received in the ICP lab as completed digestates. Samples are stored in 50 mL plastic centrifuge tubes, 100 mL digestion vessels or in 100 mL volumetric flasks.

6.2 Water samples analyzed by EPA method 200.7 are preserved after arrival at the laboratory. These samples are held for a minimum of 16 hours and the pH verified to be <2 prior to digestion.

6.3 Soil samples are diluted prior to instrumental analysis by a factor of 2. This allows the method to meet the required 1 g of sample to 200 mL dilution during digestion.

6.4 Following analysis, digestates are stored until two weeks after all results have been reviewed and then brought to 3< pH<10 and disposed of through the sewer system.

7 APPARATUS & EQUIPMENT

7.1 Inductively Coupled Plasma Atomic Emission Spectrometer

7.1.1 Thermo Jarrell Ash IRIS.

7.1.2 Thermo Scientific ICAP 6500

7.2 Concentric nebulizers.

7.3 Torches and injector tips for each ICP.

7.4 Cyclonic spray chambers for each instrument.

7.5 Water coolers for each ICP (internal on the IRIS.)

7.6 Argon Humidifiers for the IRIS and ICAP 6500.

7.7 Peristaltic Pumps for each Spectrometer.

7.8 ASX-520 autosamplers for the IRIS and ICAP 6500.

7.9 RF Generators for each ICP (internal on the IRIS and ICAP 6500).

7.10 Computer system interfaced to each ICP. A compatible Windows-based data system is used to acquire, store, and perform calculations on raw data.

8 STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

8.1 Standards Preparation

8.1.1 All standards are prepared from NIST traceable or certified standards as per SOP ADM-DATANTRY *Making Entries into Logbooks and onto Benchsheets.*. Manufacturer's expiration dates are used to determine the viability of standards.

8.1.2 Calibration Standards

Calibration standards are prepared from commercially purchased single element 1000 ppm or 10,000 ppm stock standards as well as pre-mixed multi element stock standards. All standards are aliquoted using Class A volumetric pipettes, or calibrated fixed and adjustable volume autopipetters. All dilutions are made in Class A volumetric glassware.

The standard mixes for each ICP system vary based on the requirements of each instrument. The composition of the IRIS calibration standards are outlined in Table 3. The composition of the ICAP 6500 standards are outlined in Table 4.

8.1.3 Continuing Calibration Verification (CCV) Standards

CCV standards are analyzed at the midpoint of the calibration. These standards are produced by making a two-fold dilution of each calibration standard. The CCV standards are then run in sequence during the analytical run.

8.1.4 Initial Calibration Verification (ICV) Standards

The ICV working standards are produced by direct dilution of three certified mixed stock solutions (QCP-CICV1, QCP-CICV2, and QCP-CICV3) purchased from Inorganic Ventures or another qualified vendor and various single element stock solutions from sources different than the calibration standards. The composition of these standards is outlined in Table 5.

8.1.5 Interference Check Solutions (ICSA & ICSAB)

The ICSA and ICSAB working standards are produced by direct dilution of certified mixed stock solutions (CLPP-ICS-A and CLPP-ICS-B or equivalent.) Antimony is also added to the ICSAB solution from a 1000 ppm single element stock standard. The composition of these standards is outlined in Table 6.

8.1.6 CRI/Low Level Calibration Verification

The CRI, Low Level Initial Calibration Verification (LLICV), and Low Level Continuing Calibration Verification (LLCCV) are produced by diluting 1000 or 10000ppm single stock standards into a 100X intermediate standard and then diluted 1/100 to obtain the MRL level. Note: The level used is that of the normal MRL used for both instruments.

8.1.7 The solutions and materials used for the LCS and matrix spikes are described in the applicable digestion SOP.

8.1.8 Standard Log

The analyte, source, initial volume, final volume, final concentration and expiration date are recorded in a standard logbook kept in the ICP lab. The operator who prepares the standard must date and initial the entry in the standards logbook. The operator also places his initials and the date prepared on the standard container. In addition to working standards used in calibration, all other standards used in the analytical run such as ICVs, MRL standards, and other project or client specific standards shall be documented in the standard logbook.

8.2 High Purity Argon.

8.3 Capillary, rinse and peristaltic pump tubing.

8.4 17 x 100mm polypropylene test tubes.

9 PREVENTIVE MAINTENANCE

9.1 All maintenance is documented in the instrument logbook; including torch, nebulizer, and spray chamber cleaning. All instrument filters are vacuumed monthly. Dirty ICP torches and mixing chambers are soaked in aqua regia overnight, rinsed and placed in a clean dry area. The nebulizer is back flushed with acid or DI water as needed.

9.2 IRIS-specific procedures

9.2.1 Each time the nebulizer is changed, the instrument optimization procedure is performed. A 10 ppm As standard is aspirated and the intensity counts are maximized by adjusting the depth to which the nebulizer is inserted into the spray chamber.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the SOP *ADM-TRANDOC*.

11 PROCEDURE

11.1 Operating Parameters

11.1.1 For the Thermo Jarrell Ash IRIS, the operating parameters are defined in the operating system *Method* file. Default operating parameters are given in file GALILEO. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the IRIS and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.1.2 For the Thermo Scientific ICAP 6500, the operating parameters are defined in the *Method* file. Default operating parameters are given in *Tools/Options/New Method Parameters*. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the ICAP 6500 and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.2 Calibration/Standardization

11.2.1 IRIS

11.2.1.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.1.2 An internal standard is used for routine analyses on this instrument. Scandium is used as the internal standard. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 1ug/mL.

11.2.1.3 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.1.4 The Cu/Mn ratio is calculated from Standard A. Ratio is to remain within 20% of the value when IECs were determined. This data is recorded in the instrument logbook.

11.2.2 ICAP 6500

11.2.2.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.2.2 An internal standard is used for routine analyses on this instrument. Yttrium and Indium are used as internal standards. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 2.64 ug/mL for Y, 5.28 ug/mL for In.

11.2.2.3 Run a peak check standard and adjust peaks as needed.

11.2.2.4 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.2.5 The Cu/Mn ratio is calculated from Standard A using Mn2576. Ratio is to remain within 20% of the value when IECs were determined. This data is recorded in the instrument logbook.

11.2.3 Standardization is completed by analyzing an ICV for each analyte to be determined. For method 200.7 the result must be within ± 5% of the true value. For method 6010B/C the result must be within ± 10% of the true value. If the ICV fails when running method 6010C, either the calibration standards or the ICV must be prepared fresh and the instrument re-standardized. If the ICV fails when running methods 200.7 and 6010B only re-standardization is necessary.

11.2.4 Method 6010C also requires a LLICV be analyzed at the MRL level. The result must be within ± 30% of the true value. The LLICV need not be made up with stock standards different than those of the calibration standards. A LLICV is not necessary when running methods 200.7 and 6010B.

11.3 Analytical Run

11.3.1 Following standardization and ICV analysis, the remainder of the run is determined by what analytical method is being performed. These are listed below.

11.3.1.1 CLP ILM04.0: ICB, CCV, CCB, CRI, ICSA, ICSAB, CCV, CCB, routine samples. The CRI, ICSA, and ICSAB will be analyzed every 20 samples. They will be labeled with an F indicating Final. Each set will be numbered in increasing order, i.e. ICSAF1, ICSAF2.

11.3.1.2 Methods 200.7 and 6010B/C: ICB, CCV, CCB, CRI, ICSA, ICSAB, routine samples.

11.3.2 Evaluate the initial QC using the following criteria:

11.3.2.1 For methods 200.7 and 6010B/C, the following criteria apply:

- The ICB and CCB results are evaluated using method specified requirements. The following guidelines should also be used to determine acceptability:
  - For 200.7, the result should be less than 3 times the standard deviation of the mean background signal.
  - For method 6010B, the result should be less than the Method Detection Limit (MDL). In cases where the associated sample results are being reported to the Method Reporting Limit (MRL) the result may be greater than the MDL if the result does not adversely impact data quality.
  - For method 6010C, the result should be less than the Lower Limit of Quantitation (LOQ).
  - Where project specifications allow, the result may be over the MDL if the result does not adversely impact data quality.
- The CCV immediately following standardization must verify within ± 10% of the true values with a relative standard deviation of <5% from 2 replicate integrations for methods 6010B/C. For 200.7, the first CCV must verify within ± 5% with a RSD of <3% from 4 replicates. Calculate %RSD as follows:

$$\%RSD = \frac{StdDev_{CCV}}{Average_{CCV}} x\,100$$

where: $StdDev_{ccv}$ = Standard deviation of the replicate integrations
$Average_{ccv}$ = Average of the replicate CCV integrations

- The CRI is a low level standard with concentrations at the RL. For DoD projects, the CRI standard concentrations will be equal to the project RLs and results must verify within 20% of the true value. For method 6010C the CRI results should be within 30% of the true value. For 200.7 and 6010B the CRI results should be greater than the MDL and less than 2X the MRL.

- The ICSA is run to check the validity of the Interelement Correction Factors (IECs).

Note: DoD QSM requires this to be run at the beginning of each analytical run.

- The ICSAB must be within 20% of the expected value for the CLPP-ICS-B elements and Sb.

11.3.2.2 The ICV, LLICV, ICB, CCV, CCB, CRI, and ICSAB must meet the criteria listed. Reanalyze any elements that fail.

11.3.2.3 For CLP, refer to SOW ILM04.0 for acceptance criteria.

11.3.3 Continuing Calibration Verification

11.3.3.1 CCVs are analyzed after every 10 samples and at the end of the analytical run. They must verify within ±10% of the expected value with a RSD of <10%.

11.3.3.2 CCBs are analyzed after every 10 samples and at the end of the analytical run. CCBs are evaluated as in section 11.3.2.1.

11.3.3.3 ICS solutions are to be run at least every 8 hours. Results for the spiked analytes in the ICSAB solution must agree within ±20% of the expected value.

11.3.3.4 Method 6010C requires a LLCCV be analyzed at the end of each analysis batch. The LLCCV is at the MRL level and must verify within ± 30% of the true value. Reanalyze any elements to be reported at low levels that are bracketed by the LLCCV if the standard fails.

11.3.3.5 If the CCV, CCB or ICS solutions fail, reanalyze any elements to be reported.

12 QA/QC REQUIREMENTS

12.1 Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery for each analyte must meet LCS criteria and the RSD< 30%.

12.2 Method Detection Limits

12.2.1 A Method Detection Limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates at a level near or below the MRL. Follow the procedures in Section 11 to analyze the samples. Refer to the CAS SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL.)*

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDLs must be performed whenever there is a significant change in the background or instrument response.

12.2.3 A Limit of Detection (LOD) check must be performed after establishing the MDL and at least annually (quarterly if DoD) afterward. A blank is spiked with analytes at 1-4X the MDL and carried through the preparation and analytical procedure. The LOD is verified when the signal/noise ratio is > 3 for all analytes.

12.3 Limit of Quantitation Check(LOQ)/Lower Limit of Quantitation Check(LLQC)

For Method 6010C and drinking waters by method 200.7 a Lower Limit of Quantitation Check (LOQ/LLOQ) sample must be analyzed after establishing the MRL and at least annually (quarterly if DoD) afterward to demonstrate the desired detection capability. The LOQ/LLOQ sample is spiked at 1-2X the MRL and must be carried through the entire preparation and analytical procedure. Limits of quantitation are verified when all analytes are detected within 30% of their true value.

12.4 Linear Dynamic Range

The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing at least three succeeding higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% above or below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified quarterly or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.5 Instrument Detection Limit

On a quarterly basis, the instrument detection limits for all analytes are determined as per procedures outlined in ILM04.0 (Section E, paragraph 10, 12 resp.). IDLs are determined using blanks and this data is kept on file.

12.6 Interelement Correction Factors

Semi-annually, instrument interferences are calculated as per ILM04.0 (Section E, paragraph 11) and Method 6010B/C. During the course of routine work, other interferences may be found. They are verified by the operator during the analytical run and data is manually corrected. Copies of this data are kept on file.

12.7 Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches*. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories*. General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)*. General QC Samples are:

12.7.1 Each sample preparation batch must have a method blank associated with it. The method blank result should be < MRL. If the method blank is found to be contaminated, it may be reported if the concentration in the associated samples is at least 20 times the amount found in the method blank for methods 200.7 and 6010B, otherwise redigest the batch. For Method 6010C, the method blank may be reported if the concentration in the associated samples is at least 10 times the amount found in the method blank.

Note: DoD QSM requires contamination in the MB be <1/2 the RL or < 1/10 any sample amount.

12.7.2 A Laboratory Control Sample (LCS) is digested one per batch, or per 20 samples. For method 200.7, the LCS recovery criteria is 85-115% for water samples. For method 6010B/C, the control limits are derived from lab data and are listed in Table 2. These limits should be no wider than 80-120%. For soil samples, the recovery must fall within the ranges specified for the reference material. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the LCS fails the acceptance criteria, redigest the batch of samples. For specifics on the preparation and composition of LCS samples refer to the appropriate digestion SOP.

12.7.3 A Duplicate sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analysis. The default

criteria of may be used if statistically generated criteria is broader or insufficient points are available for accurate statistical limits. Currently, statistically generated criteria are broader and the default is used for all elements but Manganese, for which the limit is 17% RPD. The RPD criteria are <30% for soil samples and <20% for water samples for methods 200.7 and 6010B. The RPD criteria is <20% for both soils and waters for method 6010C. Criteria are subject to change as statistical data are generated. If the RPD is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous).

12.7.4 A Laboratory fortified Blank (LFB) at the MRL is digested and analyzed with every batch of drinking water samples (method 200.7). The default acceptance criteria of 50-150% are to be used until sufficient data points are acquired to calculate in-house control limits.

12.7.5 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analyses. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria for method 200.7 is 70-130% for both water and soil samples. For 6010B, the control limits are derived from lab data and are listed in Table 2. For 6010C, Table 2 is used unless the control limits are broader than the default criteria of 75-125% in which case the default criteria are used. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the recovery is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous). If the sample concentration is >4x the spike level, no action is required and data is flagged accordingly. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

12.7.6 The analyst must run a dilution test (1:5 dilution) to test for matrix interference if there is a reason to suspect such an interference and for all tier III deliverables when running methods 200.7 and 6010B. For Method 6010C, a dilution test must be run on each preparation batch and agree within ± 10% of the original result.

12.7.7 If the analyte concentration is too low to permit a dilution test, then a post digestion spike may be performed. For Method 6010C, a post-spike is performed on all preparation batches. This spike should be recovered to within 80-120% of the true value.

Note: DoD QSM recovery acceptance limits are 75-125%.

12.7.8 Additional QC measures include control charting and compiling of QC data for generation of control limits.

12.8 CLP analyses are performed as per the QA/QC guidelines in the most current CLP SOW.

13 DATA REDUCTION, REVIEW, AND REPORTING

13.1 Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result. The wavelengths used to quantify each metal are summarized in Table 7 for the IRIS and Table 8 for the ICAP6500.

Aqueous samples are reported in µg/L:

$$\mu g/L\,(Sample) = C^{*} \times Digestion\,Dilution\,Factor \times Post\,Digestion\,Dilution\,Factor \times 1000 \mu g/mg$$

Solid samples are reported in mg/Kg:

$$mg/Kg\,(Sample) = C^{*} \times Post\,Digestion\,Dilution\,Factor \times \frac{Digestion\,Vol\,(ml)}{Sample\,wt.\,(g)} \times \frac{1L}{1000ml} \times \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L.

13.2 If total hardness is to be reported, use Calcium and Magnesium results to calculate as follows. For reporting calcium hardness, use only the calcium portion of the equation.

$$Hardness, mg\ equivalent\ CaCO_3/L = 2.497[Ca, mg/L] + 4.118[Mg, mg/L]$$

13.3 A daily run log of all samples analyzed is maintained. All CLP data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.4 Data Review and Reporting

13.4.1 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12. The data is then placed in a work order file until complete. When the work order is complete, a report is generated. A final review is performed and the data is delivered to the project management department.

14 CORRECTIVE ACTION

14.1 Refer to the SOP for *Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2 Handling out-of-control or unacceptable data

14.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2 Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when:

- Corrective action is not taken or not possible
- Corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis.
- Reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.1 The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL)*. Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16 POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible and within method requirements. Standards are prepared in volumes consistent with the laboratory use in order to minimize the volume of expired standards to be disposed. The threat to the environment from reagents used in this method may be minimized when recycled or disposed of properly.

17 WASTE MANAGEMENT

17.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2 This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 3-10 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18 TRAINING

18.1 Refer to the *SOP for Documentation of Training* for standard procedures.

18.2 Training outline

18.2.1 Review literature (see references section). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.2.2 The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of approximately two weeks. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.2.3 Perform initial precision and recovery (IPR) study as described in Section 12.1 for water samples. Summaries of the IPR are reviewed and signed by the supervisor.

18.3 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

19 CHANGES SINCE THE LAST REVISION

19.1 Instrumentation no longer in service removed from SOP.
19.2 Sec 1.1 removed 6010B and included in sec 1.3.
19.3 Sec 1.3 is new.
19.4 Sec 12.7 is updated.
19.5 Sec 14 Updated.
19.6 Sec 12.2.1 and Sec 15 updated SOP reference

20 REFERENCES

20.1 USEPA, Contract Laboratory Program, SOW #ILM04.0

20.2 Thermo Jarrell Ash ICAP61 Manual

20.3 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010B, Revision 2, December 1996.

20.4 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010C, Revision 3, February 2007.

20.5 USEPA, Methods for Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, Method 200.7, Revision 4.4, May 1994.

20.6 *Hardness by Calculation, Method 2340B,* Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

TABLE 1

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Water	CLP	50	30	ug/L	6010 / 200.7
Antimony	Water	CLP	50	30	ug/L	6010 / 200.7
Arsenic	Water	CLP	100	20	ug/L	6010 / 200.7
Barium	Water	CLP	5	0.7	ug/L	6010 / 200.7
Beryllium	Water	CLP	5	0.4	ug/L	6010 / 200.7
Boron	Water	CLP	50	9	ug/L	6010 / 200.7
Cadmium	Water	CLP	5	3	ug/L	6010 / 200.7
Calcium	Water	CLP	50	30	ug/L	6010 / 200.7
Chromium	Water	CLP	5	3	ug/L	6010 / 200.7
Cobalt	Water	CLP	10	3	ug/L	6010 / 200.7
Copper	Water	CLP	10	7	ug/L	6010 / 200.7
Iron	Water	CLP	20	3	ug/L	6010 / 200.7
Lead	Water	CLP	50	20	ug/L	6010 / 200.7
Lithium	Water	CLP	10	6	ug/L	6010 / 200.7
Magnesium	Water	CLP	20	0.7	ug/L	6010 / 200.7
Manganese	Water	CLP	5	0.6	ug/L	6010 / 200.7
Molybdenum	Water	CLP	10	6	ug/L	6010 / 200.7
Nickel	Water	CLP	20	20	ug/L	6010 / 200.7
Phosphorus	Water	CLP	200	200	ug/L	6010 / 200.7
Potassium	Water	CLP	2000	900	ug/L	6010 / 200.7
Selenium	Water	CLP	100	40	ug/L	6010 / 200.7
Silicon	Water	CLP	400	40	ug/L	6010 / 200.7
Silver	Water	CLP	10	6	ug/L	6010 / 200.7
Sodium	Water	CLP	100	50	ug/L	6010 / 200.7
Strontium	Water	CLP	10	0.5	ug/L	6010 / 200.7
Thallium	Water	CLP	200	50	ug/L	6010 / 200.7
Tin	Water	CLP	50	20	ug/L	6010 / 200.7
Titanium	Water	CLP	10	3	ug/L	6010 / 200.7
Vanadium	Water	CLP	10	5	ug/L	6010 / 200.7
Zinc	Water	CLP	10	7	ug/L	6010 / 200.7

TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Water	3010A	50	50	ug/L	6010
Antimony	Water	3010A	50	20	ug/L	6010
Arsenic	Water	3010A	100	30	ug/L	6010
Barium	Water	3010A	5	2	ug/L	6010
Beryllium	Water	3010A	5	0.2	ug/L	6010
Boron	Water	3010A	50	30	ug/L	6010
Cadmium	Water	3010A	5	3	ug/L	6010
Calcium	Water	3010A	50	10	ug/L	6010
Chromium	Water	3010A	5	2	ug/L	6010
Cobalt	Water	3010A	10	4	ug/L	6010
Copper	Water	3010A	10	6	ug/L	6010
Iron	Water	3010A	20	3	ug/L	6010
Lead	Water	3010A	50	30	ug/L	6010
Lithium	Water	3010A	10	8	ug/L	6010
Magnesium	Water	3010A	20	0.5	ug/L	6010
Manganese	Water	3010A	5	2	ug/L	6010
Molybdenum	Water	3010A	10	4	ug/L	6010
Nickel	Water	3010A	20	20	ug/L	6010
Phosphorus	Water	3010A	200	90	ug/L	6010
Potassium	Water	3010A	2000	1000	ug/L	6010
Selenium	Water	3010A	100	40	ug/L	6010
Silicon	Water	3010A	400	200	ug/L	6010
Silver	Water	3010A	10	9	ug/L	6010
Sodium	Water	3010A	100	60	ug/L	6010
Strontium	Water	3010A	10	0.5	ug/L	6010
Thallium	Water	3010A	200	60	ug/L	6010
Tin	Water	3010A	50	20	ug/L	6010
Titanium	Water	3010A	10	2	ug/L	6010
Vanadium	Water	3010A	10	4	ug/L	6010
Zinc	Water	3010A	10	3	ug/L	6010

TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Soil	3050B	10	7	mg/Kg	6010 / 200.7
Antimony	Soil	3050B	10	8	mg/Kg	6010 / 200.7
Arsenic	Soil	3050B	20	5	mg/Kg	6010 / 200.7
Barium	Soil	3050B	2	0.2	mg/Kg	6010 / 200.7
Beryllium	Soil	3050B	1	0.03	mg/Kg	6010 / 200.7
Boron	Soil	3050B	10	2	mg/Kg	6010 / 200.7
Cadmium	Soil	3050B	1	1	mg/Kg	6010 / 200.7
Calcium	Soil	3050B	10	2	mg/Kg	6010 / 200.7
Chromium	Soil	3050B	2	0.7	mg/Kg	6010 / 200.7
Cobalt	Soil	3050B	2	0.6	mg/Kg	6010 / 200.7
Copper	Soil	3050B	2	0.7	mg/Kg	6010 / 200.7
Iron	Soil	3050B	4	0.6	mg/Kg	6010 / 200.7
Lead	Soil	3050B	20	7	mg/Kg	6010 / 200.7
Magnesium	Soil	3050B	4	2	mg/Kg	6010 / 200.7
Manganese	Soil	3050B	2	0.07	mg/Kg	6010 / 200.7
Molybdenum	Soil	3050B	2	0.9	mg/Kg	6010 / 200.7
Nickel	Soil	3050B	4	3	mg/Kg	6010 / 200.7
Phosphorus	Soil	3050B	40	20	mg/Kg	6010 / 200.7
Potassium	Soil	3050B	200	200	mg/Kg	6010 / 200.7
Selenium	Soil	3050B	40	6	mg/Kg	6010 / 200.7
Silver	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Sodium	Soil	3050B	20	20	mg/Kg	6010 / 200.7
Strontium	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Thallium	Soil	3050B	40	20	mg/Kg	6010 / 200.7
Tin	Soil	3050B	10	5	mg/Kg	6010 / 200.7
Titanium	Soil	3050B	2	0.5	mg/Kg	6010 / 200.7
Vanadium	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Zinc	Soil	3050B	2	0.9	mg/Kg	6010 / 200.7

TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL (1)	MDL (1)	Units	Method
Aluminum	Tissue	PSEP	10	6	mg/Kg	6010
Antimony	Tissue	PSEP	5	3	mg/Kg	6010
Barium	Tissue	PSEP	1	0.2	mg/Kg	6010
Beryllium	Tissue	PSEP	1	0.02	mg/Kg	6010
Boron	Tissue	PSEP	5	0.6	mg/Kg	6010
Cadmium	Tissue	PSEP	0.5	0.3	mg/Kg	6010
Calcium	Tissue	PSEP	40	20	mg/Kg	6010
Chromium	Tissue	PSEP	1	0.6	mg/Kg	6010
Cobalt	Tissue	PSEP	1	0.4	mg/Kg	6010
Copper	Tissue	PSEP	1	0.5	mg/Kg	6010
Iron	Tissue	PSEP	4	4	mg/Kg	6010
Lead	Tissue	PSEP	5	3	mg/Kg	6010
Magnesium	Tissue	PSEP	4	3	mg/Kg	6010
Manganese	Tissue	PSEP	0.5	0.2	mg/Kg	6010
Molybdenum	Tissue	PSEP	1	0.7	mg/Kg	6010
Nickel	Tissue	PSEP	2	1	mg/Kg	6010
Phosphorus	Tissue	PSEP	20	10	mg/Kg	6010
Potassium	Tissue	PSEP	200	200	mg/Kg	6010
Sodium	Tissue	PSEP	10	8	mg/Kg	6010
Strontium	Tissue	PSEP	1	0.2	mg/Kg	6010
Tin	Tissue	PSEP	5	2	mg/Kg	6010
Vanadium	Tissue	PSEP	1	0.4	mg/Kg	6010
Zinc	Tissue	PSEP	1	0.4	mg/Kg	6010

(1) Dry Weight Basis

TABLE 2

QC Limits

Element	Matrix	Digestion	Method	6010		200.7		RPD
				LCS Limit	MS Limit	LCS Limit	MS Limit	Limit
Aluminum	Water	CLP	6010 / 200.7	93-111	83-119	85-115	70-130	20
Antimony	Water	CLP	6010 / 200.7	95-111	82-119	85-115	70-130	20
Arsenic	Water	CLP	6010 / 200.7	94-113	84-116	85-115	70-130	20
Barium	Water	CLP	6010 / 200.7	93-114	76-127*	85-115	70-130	20
Beryllium	Water	CLP	6010 / 200.7	93-112	82-116	85-115	70-130	20
Boron	Water	CLP	6010 / 200.7	89-112	76-132*	85-115	70-130	20
Cadmium	Water	CLP	6010 / 200.7	92-116	71-145*	85-115	70-130	20
Calcium	Water	CLP	6010 / 200.7	94-111	75-125	85-115	70-130	20
Chromium	Water	CLP	6010 / 200.7	95-113	85-119	85-115	70-130	20
Cobalt	Water	CLP	6010 / 200.7	94-113	84-119	85-115	70-130	20
Copper	Water	CLP	6010 / 200.7	93-112	83-115	85-115	70-130	20
Iron	Water	CLP	6010 / 200.7	94-113	58-142*	85-115	70-130	20
Lead	Water	CLP	6010 / 200.7	94-114	81-122	85-115	70-130	20
Lithium	Water	CLP	6010 / 200.7	85-115	70-130*	85-115	70-130	20
Magnesium	Water	CLP	6010 / 200.7	91-112	75-125	85-115	70-130	20
Manganese	Water	CLP	6010 / 200.7	95-112	82-122	85-115	70-130	20
Molybdenum	Water	CLP	6010 / 200.7	93-114	78-126*	85-115	70-130	20
Nickel	Water	CLP	6010 / 200.7	93-115	82-122	85-115	70-130	20
Phosphorus	Water	CLP	6010 / 200.7	85-115	70-130*	85-115	70-130	20
Potassium	Water	CLP	6010 / 200.7	89-117	75-125	85-115	70-130	20
Selenium	Water	CLP	6010 / 200.7	92-112	79-120	85-115	70-130	20
Silicon	Water	CLP	6010 / 200.7	85-115	70-130*	85-115	70-130	20
Silver	Water	CLP	6010 / 200.7	94-111	80-119	85-115	70-130	20
Sodium	Water	CLP	6010 / 200.7	92-116	75-125	85-115	70-130	20
Strontium	Water	CLP	6010 / 200.7	85-115	70-130*	85-115	70-130	20
Thallium	Water	CLP	6010 / 200.7	88-120	62-128*	85-115	70-130	20
Tin	Water	CLP	6010 / 200.7	80-120	75-125	85-115	70-130	20
Titanium	Water	CLP	6010 / 200.7	85-115	70-130*	85-115	70-130	20
Vanadium	Water	CLP	6010 / 200.7	94-110	87-116	85-115	70-130	20
Zinc	Water	CLP	6010 / 200.7	94-111	83-117	85-115	70-130	20

Where recovery limits are greater than 6010B limits or not calculated due to insufficient data points, the required method limits are used.

* 6010C default limits are 75-125 and are to be used when the calculated limits are greater than 75-125.

TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	LCS Limit	MS Limit	RPD Limit
Aluminum	Water	3010A	6010	93-111	83-119	20
Antimony	Water	3010A	6010	95-111	82-119	20
Arsenic	Water	3010A	6010	94-113	84-116	20
Barium	Water	3010A	6010	93-114	76-127*	20
Beryllium	Water	3010A	6010	93-112	82-116	20
Boron	Water	3010A	6010	89-112	76-132*	20
Cadmium	Water	3010A	6010	92-116	71-145*	20
Calcium	Water	3010A	6010	94-111	75-125	20
Chromium	Water	3010A	6010	95-113	85-119	20
Cobalt	Water	3010A	6010	94-113	84-119	20
Copper	Water	3010A	6010	93-112	83-115	20
Iron	Water	3010A	6010	94-113	58-142*	20
Lead	Water	3010A	6010	94-114	81-122	20
Lithium	Water	3010A	6010	85-115	70-130*	20
Magnesium	Water	3010A	6010	91-112	75-125	20
Manganese	Water	3010A	6010	95-112	82-122	20
Molybdenum	Water	3010A	6010	93-114	78-126*	20
Nickel	Water	3010A	6010	93-115	82-122	20
Phosphorus	Water	3010A	6010	85-115	70-130*	20
Potassium	Water	3010A	6010	89-117	75-125	20
Selenium	Water	3010A	6010	92-112	79-120	20
Silicon	Water	3010A	6010	85-115	70-130*	20
Silver	Water	3010A	6010	94-111	80-119	20
Sodium	Water	3010A	6010	92-116	75-125	20
Strontium	Water	3010A	6010	85-115	70-130*	20
Thallium	Water	3010A	6010	88-120	62-128*	20
Tin	Water	3010A	6010	80-120	75-125	20
Titanium	Water	3010A	6010	85-115	70-130*	20
Vanadium	Water	3010A	6010	94-110	87-116	20
Zinc	Water	3010A	6010	94-111	83-117	20

Where recovery limits are greater than 6010B limits or not calculated due to insufficient data points, the required limits are used.

* 6010C default limits are 75-125 and are to be used when the calculated limits are greater than 75-125.

TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	6010		200.7		RPD
				LCS Limit	MS Limit	LCS Limit	MS Limit	Limit
Aluminum	Soil	3050B	6010 / 200.7	61-152	75-125	61-152	70-130	20
Antimony	Soil	3050B	6010 / 200.7	36-147	10-128*	36-147	70-130	20
Arsenic	Soil	3050B	6010 / 200.7	81-123	49-139*	81-123	70-130	20
Barium	Soil	3050B	6010 / 200.7	77-139	79-125	77-139	70-130	20
Beryllium	Soil	3050B	6010 / 200.7	88-124	82-116	88-124	70-130	20
Boron	Soil	3050B	6010 / 200.7	37-174	33-164*	37-174	70-130	20
Cadmium	Soil	3050B	6010 / 200.7	92-125	58-144*	92-125	70-130	20
Calcium	Soil	3050B	6010 / 200.7	80-131	75-125	80-131	70-130	20
Chromium	Soil	3050B	6010 / 200.7	93-123	22-184*	93-123	70-130	20
Cobalt	Soil	3050B	6010 / 200.7	78-134	84-114	78-134	70-130	20
Copper	Soil	3050B	6010 / 200.7	85-118	51-147*	85-118	70-130	20
Iron	Soil	3050B	6010 / 200.7	64-154	75-125	64-154	70-130	20
Lead	Soil	3050B	6010 / 200.7	82-131	49-148*	82-131	70-130	20
Magnesium	Soil	3050B	6010 / 200.7	83-130	75-125	83-130	70-130	20
Manganese	Soil	3050B	6010 / 200.7	83-130	25-178*	83-130	70-130	20
Molybdenum	Soil	3050B	6010 / 200.7	84-134	75-116	84-134	70-130	20
Nickel	Soil	3050B	6010 / 200.7	92-123	74-126*	92-123	70-130	20
Phosphorus	Soil	3050B	6010 / 200.7	85-115**	75-125	85-115**	70-130	20
Potassium	Soil	3050B	6010 / 200.7	80-131	75-125	80-131	70-130	20
Selenium	Soil	3050B	6010 / 200.7	68-166	68-127*	68-166	70-130	20
Silver	Soil	3050B	6010 / 200.7	90-118	44-138*	90-118	70-130	20
Sodium	Soil	3050B	6010 / 200.7	87-121	75-125	87-121	70-130	20
Strontium	Soil	3050B	6010 / 200.7	80-120	75-125	80-120	70-130	20
Thallium	Soil	3050B	6010 / 200.7	27-171	25-153*	27-171	70-130	20
Tin	Soil	3050B	6010 / 200.7	80-120	75-125	80-120	70-130	20
Titanium	Soil	3050B	6010 / 200.7	40-160	75-125	40-160	70-130	20
Vanadium	Soil	3050B	6010 / 200.7	88-121	77-122	88-121	70-130	20
Zinc	Soil	3050B	6010 / 200.7	88-126	32-168*	88-126	70-130	20

* 6010C default limits are 75-125 and are to be used when the calculated limits are greater than 75-125.
** Blank Spike limit. No value available for D045540.
LCS Material: ERA PrioityPollutant/CLP Soils Lot D045540

TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	LCS Limit [2]	MS* Limit	RPD Limit [2]
Aluminum	Tissue	PSEP	6010	85-115	70-130	30
Antimony	Tissue	PSEP	6010	85-115	70-130	30
Barium	Tissue	PSEP	6010	85-115	70-130	30
Beryllium	Tissue	PSEP	6010	85-115	70-130	30
Boron	Tissue	PSEP	6010	85-115	70-130	30
Cadmium	Tissue	PSEP	6010	85-115	70-130	30
Calcium	Tissue	PSEP	6010	85-115	70-130	30
Chromium	Tissue	PSEP	6010	85-115	70-130	30
Cobalt	Tissue	PSEP	6010	85-115	70-130	30
Copper	Tissue	PSEP	6010	85-115	70-130	30
Iron	Tissue	PSEP	6010	85-115	70-130	30
Lead	Tissue	PSEP	6010	85-115	70-130	30
Magnesium	Tissue	PSEP	6010	85-115	70-130	30
Manganese	Tissue	PSEP	6010	85-115	70-130	30
Molybdenum	Tissue	PSEP	6010	85-115	70-130	30
Nickel	Tissue	PSEP	6010	85-115	70-130	30
Phosphorus	Tissue	PSEP	6010	85-115	70-130	30
Potassium	Tissue	PSEP	6010	85-115	70-130	30
Sodium	Tissue	PSEP	6010	85-115	70-130	30
Strontium	Tissue	PSEP	6010	85-115	70-130	30
Tin	Tissue	PSEP	6010	85-115	70-130	30
Vanadium	Tissue	PSEP	6010	85-115	70-130	30
Zinc	Tissue	PSEP	6010	85-115	70-130	30

[1] Dry Weight Basis
[2] List limits are for "Blank Spike" analysis. SRM samples have separate limits.
*6010C requires MS Limit of 75-125.

TABLE 3
Standard A for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	10	1000	1.0
Antimony	Elemental Stock	1000	4	1000	4.0
Arsenic	(1)	100	10	1000	1.0
Arsenic	Elemental Stock	1000	4	1000	4.0
Boron	(1)	100	10	1000	1.0
Cadmium	(1)	100	10	1000	1.0
Calcium	(1)	100	10	1000	1.0
Calcium	Elemental Stock	10000	0.4	1000	4.0
Chromium	(1)	100	10	1000	1.0
Cobalt	(1)	100	10	1000	1.0
Copper	(1)	100	10	1000	1.0
Iron	(1)	100	10	1000	1.0
Lead	(1)	100	10	1000	1.0
Lead	Elemental Stock	1000	4	1000	4.0
Magnesium	(1)	100	10	1000	1.0
Magnesium	Elemental Stock	1000	3	1000	3.0
Manganese	(1)	100	10	1000	1.0
Manganese	Elemental Stock	1000	1	1000	1.0
Molybdenum	(1)	100	10	1000	1.0
Molybdenum	Elemental Stock	1000	1	1000	1.0
Nickel	(1)	100	10	1000	1.0
Selenium	(1)	100	10	1000	1.0
Selenium	Elemental Stock	1000	4	1000	4.0
Silver	(1)	100	10	1000	1.0
Tin	Elemental Stock	1000	5	1000	5.0
Thallium	(1)	100	10	1000	1.0
Thallium	Elemental Stock	1000	9	1000	9.0
Titanium	(1)	100	10	1000	1.0
Vanadium	(1)	100	10	1000	1.0
Zinc	(1)	100	10	1000	1.0
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

TABLE 3 continued
Standard B for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	1	1000	10
Barium	Elemental Stock	10000	0.5	1000	5.0
Beryllium	Elemental Stock	1000	0.1	1000	0.1
Calcium	Elemental Stock	10000	5	1000	50
Iron	Elemental Stock	10000	5	1000	50
Lithium	Elemental Stock	1000	1	1000	1.0
Magnesium	Elemental Stock	10000	5	1000	50
Manganese	Elemental Stock	1000	20	1000	20
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	0.5	1000	5
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	1	1000	1.0
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

TABLE 4
Standard A for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	5	1000	0.5
Beryllium	(1)	100	5	1000	0.5
Boron	(1)	100	5	1000	0.5
Cadmium	(1)	100	5	1000	0.5
Calcium	(1)	100	5	1000	0.5
Chromium	(1)	100	5	1000	0.5
Cobalt	(1)	100	5	1000	0.5
Copper	(1)	100	5	1000	0.5
Iron	(1)	100	5	1000	0.5
Lead	(1)	100	5	1000	0.5
Magnesium	(1)	100	5	1000	0.5
Manganese	(1)	100	5	1000	0.5
Molybdenum	(1)	100	5	1000	0.5
Nickel	(1)	100	5	1000	0.5
Selenium	(1)	100	5	1000	0.5
Silver	(1)	100	5	1000	0.5
Tin	Elemental Stock	1000	0.5	1000	0.5
Thallium	(1)	100	5	1000	0.5
Titanium	(1)	100	5	1000	0.5
Vanadium	(1)	100	5	1000	0.5
Zinc	(1)	100	5	1000	0.5
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

TABLE 4 continued
Standard B for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	2	1000	20
Arsenic	Elemental Stock	1000	2	1000	2
Barium	Elemental Stock	10000	2	1000	20
Calcium	Elemental Stock	10000	2	1000	20
Iron	Elemental Stock	10000	2	1000	20
Lithium	Elemental Stock	1000	2	1000	2
Magnesium	Elemental Stock	10000	2	1000	20
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	2	1000	20
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	2	1000	2
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

TABLE 5
ICP ICV Standards

ICV1 Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	QCP-CICV-1	1000	2.5	500	5.0
Antimony	QCP-CICV-2	500	2.5	500	2.5
Arsenic	QCP-CICV-3	500	2.5	500	2.5
Barium	QCP-CICV-1	1000	2.5	500	5.0
Beryllium	QCP-CICV-1	25	2.5	500	0.125
Cadmium	QCP-CICV-3	250	2.5	500	1.25
Calcium	QCP-CICV-1	2500	2.5	500	12.5
Chromium	QCP-CICV-1	100	2.5	500	0.5
Cobalt	QCP-CICV-1	250	2.5	500	1.25
Copper	QCP-CICV-1	125	2.5	500	0.625
Iron	QCP-CICV-1	500	2.5	500	2.5
Lead	QCP-CICV-3	500	2.5	500	2.5
Magnesium	QCP-CICV-1	2500	2.5	500	12.5
Manganese	QCP-CICV-1	250	2.5	500	1.25
Molybdenum	Elemental Stock	1000	1.0	500	2.0
Nickel	QCP-CICV-1	250	2.5	500	1.25
Potassium	QCP-CICV-1	2500	2.5	500	12.5
Selenium	QCP-CICV-3	500	2.5	500	2.5
Silver	QCP-CICV-1	125	2.5	500	0.625
Sodium	QCP-CICV-1	2500	2.5	500	12.5
Thallium	QCP-CICV-3	500	2.5	500	2.5
Titanium	Elemental Stock	1000	1.0	500	2.0
Vanadium	QCP-CICV-1	250	2.5	500	1.25
Zinc	QCP-CICV-1	250	2.5	500	1.25
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

TABLE 5 continued

ICVB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	1000	0.5	500	1
Boron	Elemental Stock	1000	2.5	500	5
Calcium	Elemental Stock	1000	2.5	500	5
Iron	Elemental Stock	1000	12.5	500	25
Lithium	Elemental Stock	1000	2.5	500	5
Magnesium	Elemental Stock	1000	2.5	500	5
Manganese	Elemental Stock	1000	6.25	500	12.5
Phosphorus	Elemental Stock	1000	2.5	500	5
Silicon	Elemental Stock	1000	2.5	500	5
Strontium	Elemental Stock	1000	1	500	2
Tin	Elemental Stock	1000	2.5	500	5
Hydrochloric Acid	-		25	500	5%
Nitric Acid	-		5	500	1%

TABLE 6
ICP Interference Check Solutions

ICSA Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Calcium	CLPP-ICS-A	5000	50	500	500
Iron	CLPP-ICS-A	2000	50	500	200
Magnesium	CLPP-ICS-A	5000	50	500	500
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

ICSAB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Antimony	Elemental Stock	1000	0.5	500	1
Barium	CLPP-ICS-B	50	5	500	0.5
Beryllium	CLPP-ICS-B	50	5	500	0.5
Cadmium	CLPP-ICS-B	100	5	500	1
Calcium	CLPP-ICS-A	5000	50	500	500
Chromium	CLPP-ICS-B	50	5	500	0.5
Cobalt	CLPP-ICS-B	50	5	500	0.5
Copper	CLPP-ICS-B	50	5	500	0.5
Iron	CLPP-ICS-A	2000	50	500	200
Lead	CLPP-ICS-B	100	5	500	1
Magnesium	CLPP-ICS-A	5000	50	500	500
Manganese	CLPP-ICS-B	50	5	500	0.5
Nickel	CLPP-ICS-B	100	5	500	1
Silver	CLPP-ICS-B	100	5	500	1
Vanadium	CLPP-ICS-B	50	5	500	0.5
Zinc	CLPP-ICS-B	100	5	500	1
HCl	-	-	25	500	0.05
HNO3	-	-	5	500	0.01

TABLE 7
IRIS Analytical Wavelengths

Analyte	Wavelength	
Aluminum	237.3	
Antimony	206.8	
Arsenic	189.0	
Barium	233.5	
Beryllium	313.0	
Boron	249.7	
Cadmium	226.5	
Calcium	317.9	
Calcium	211.2	High Line
Chromium	267.7	
Cobalt	228.6	
Copper	324.7	
Iron	259.9	
Iron	271.4	High Line
Lead	220.3	
Lithium	670.7	
Magnesium	279.5	
Magnesium	202.5	High Line
Manganese	257.6	
Manganese	293.9	High Line
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

TABLE 8
ICAP 6500 Analytical Wavelengths

Analyte	Wavelength	
Aluminum	167.0	Low Line
Aluminum	394.4	
Antimony	206.8	
Antimony	217.5	Alternate
Arsenic	189.0	
Barium	455.4	
Beryllium	234.8	
Boron	249.6	
Cadmium	226.5	
Calcium	315.8	
Calcium	393.3	Low Line
Chromium	267.7	
Cobalt	230.7	
Copper	327.3	
Iron	259.9	
Lead	220.3	
Lithium	670.7	
Magnesium	279.0	High Line
Magnesium	279.5	Low Line
Magnesium	285.2	
Manganese	257.6	
Manganese	260.5	High Line
Molybdenum	202.0	
Nickel	221.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	588.9	Alternate
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	336.1	
Vanadium	292.4	
Zinc	206.2	

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

EPA Method 8270C

SOC-8270C
Revision 12
June 26, 2009

Approved By:

Supervisor		7/1/09 Date
QA Manager		7/9/09 Date
Laboratory Manager		7/9/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS
Method 8270C

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water and soil using EPA Method 8270C. This procedure may also be applicable to various miscellaneous waste samples. Tables 1 and 1A indicate compounds that may be determined by this method and lists their method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) will vary depending on the instrument used and preparation method and may also change slightly as MDL studies are updated.

1.2. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. Other compounds than those listed in Tables 1 and 1A may be analyzed. Refer to Section 1 of method 8270C.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. A 1.0 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration and the

chromatography for this compound is poor. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, to a chemical reaction in acetone, and can undergo photochemical decomposition. N-nitroso-dimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitroso-diphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) followed by initial calibration standard(s). Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP injection time.

3.2. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.3. **Standard Curve** - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.4. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.5. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.6. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.7. **Independent Calibration Verification Standard (ICV)** - A mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be purchased pre-cleaned. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum

foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction.

6.4. Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. Extracts are stored at -10°C.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2. Column: ZB-5MS with Guardian - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column. Catalog #7HG-G010-11-GGA, or equivalent.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 2 when 1.0 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic

program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2. Stock Standard Solutions (See Table 3)

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 1000 ug/ml or more. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8.2.2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10°C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards or samples indicates a problem.

8.3. Internal Standard Solutions (See Table 3) - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 4 for corresponding compounds). The nominal concentration of the standard is 4000 ng/µL. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations.

8.4. GC/MS Tuning Standard (See Table 3) - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of benzidine, DDT, and pentachlorophenol, to verify injection port inertness and GC column performance. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards (See Table 3)

8.5.1. A minimum of five initial calibration standards should be prepared from stock solutions. One of the calibration standards should be at a concentration at or below the method reporting limit; the others should correspond to the range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. At least one calibration standard must be at a concentration corresponding to a sample concentration meeting project-specific data quality objectives. Each standard should contain each analyte for detection by this method. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.5.2. The daily calibration standard (CCV) is prepared at a nominal 50 ng/µL concentration from stock solutions. The CCV is prepared weekly and can be stored at 4°C ± 2°C, or as recommended by the manufacturer. The DFTPP standard may be combined with this standard (maintaining 50 ng/µL concentration) providing tuning verification and calibration verification can be done without interferences.

8.6. QC Standards (See Table 4)

8.6.1. Surrogates: Prepare a working solution in methanol containing 2-fluorophenol, phenol-d6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14 at 100 ng/µL. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2. Matrix Spike Standards: Prepare a working solution in methanol containing all target analytes to be reported ("full list spike") at 100 ng/µL. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

Note: The spiking level of surrogate and spike may need to be adjusted according to project requirements, if dilutions are expected due to high levels of extracted components, or if a lower calibration range is used.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria given in section 11.

9.4.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the Sop for Documentation of Training, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples

11.1.1.1. Water samples are prepared using continuous liquid-liquid extraction and EPA method 3520C. Refer to the CAS SOP EXT-3520. In some circumstances, such as rush samples or for TCLP leachates, samples may be prepared using separatory funnel procedures (EPA 3510C). Refer to the CAS SOP EXT-3510.

11.1.1.2. Perform the extraction on a 1000mL aliquot of sample. For TCLP leachates, use 100mL of sample.

11.1.2. Soil, sediment, and solid samples are prepared using the either automated soxhlet extraction (SOP EXT-3541) or ultrasonic extraction (SOP EXT-3550). The nominal sample size is 30g. Sample amounts may be decreased in the case of high-concentration waste samples.

11.1.3. Product samples are prepared using EPA method 3580.

11.1.4. Extracts may be screened by GC/FID (SOP SOC-SCR). Cleanup by GPC is optional.

11.1.5. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. The recommended GC/MS operating conditions are listed below. The GC conditions may be modified to accommodate specific instrument models and configurations.

Mass range:	35-500 amu		
Scan Time:	1 sec/scan		
Initial temperature:	40°C, hold for 4 minutes		
Temperature program:	40-270°C at 10°C/min		
Final temperature:	300°C, hold until benzo[g,h,i]perylene has eluted		
Injector temperature:	250-300°C		
Detector interface temp:	300°C		
Injector:	Atas Optic 2, split method		
Final time:	32.0 min.		
Equilibration time:	0.20 min.		
Temperature ramp:	Initial temp	150°C	
	Ramp rate	8°C/sec.	
	Final temp	300°C	
Pressure profile:	Transfer pressure	10 psi	
	Transfer time	1.50 min.	
	Initial pressure	8.83 psi	
	Final pressure	38.50 psi	
Split flow:	3.0mL/min. at initial		
Sample volume:	1.0 µL		
Carrier gas:	helium at 35 cm/sec		

NOTE: The default calibration procedures are given in the following sections and, at the time of this revision, follow Method 8000B due to inconsistent implementation of 8000C across various states/agencies. The analyst should also refer to the CAS *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOC-CAL)*, where calibration procedures and policies are described. The calibration procedure(s) and options chosen must follow the CAS protocols.

NOTE: Certain state or program protocols have specific procedures for calibration. This may include all or part of Method 8000C. The analyst must ensure that the correct procedures are used. Known exceptions to 8000B are as follows:

- Arizona DHS requires the use of Method 8000C calibration procedures for projects originating in Arizona and under the AZ DHS lab licensure. Refer to the AZDHS calibration guidance document at the following online location: http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf.
- The use of quadratic regression calibration is not allowed for projects (samples) originating from South Carolina and under the SC DHEC lab certification.

Note: The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects under the DoD QSM protocols. When working on these projects all analytes must pass the %RSD criteria as stated in the method and the average %RSD cannot be used.

11.3. Initial Calibration

11.3.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

11.3.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 2 (see 8270C, Section 7.3.1 for guidance). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. To assess column performance and injection port inertness, pentachlorophenol and benzidine should be present at an acceptable level and peak tailing should not be excessive. DDT degradation should not exceed 20%. If excessive tailing, poor chromatography, or degradation of >20% is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria can not be met, the source may need cleaning, filaments replaced or other maintenance.

11.3.3. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 5 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 1 of EPA 8270C). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.3.4. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1 of EPA 8270C).

11.3.5. Establish the retention time window position for each analyte and surrogate using the midpoint standard of the initial calibration curve.

11.3.6. Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.3.7. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compound (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. The SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. If they are not acceptable, perform GC maintenance (see section 9.3).

11.3.8. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see below) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RSD = relative standard deviation.
$\overline{RF}$ = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

Calibration Check Compounds (CCC):

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
(N-Nitroso)-diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

11.3.9. If the % RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.3.10. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.3.11. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

- The mean of the RSD values for all analytes in the calibration is < 15%.
- The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.
- The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

Note: The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects under the DoD QSM protocols. When working on these projects all analytes must pass the %RSD criteria as stated in the method and the average %RSD cannot be used.

11.3.12. If all of the conditions in Section 11.3.9 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.3.8.

11.3.13. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Organics Calibration SOP.

Note: DoD project acceptance criteria is ±20% of true value.

11.4. Continuing Calibration

11.4.1. A calibration standard, or standards, at mid-concentration (See Table 3) containing all semivolatile analytes, DFTPP, and all required surrogates, must be analyzed every 12 hours during analysis.

11.4.2. The DFTPP must result in a mass spectrum (see 8270C, Section 7.3.1 for guidance) that meets the criteria given in Table 2. These criteria must be demonstrated during each 12 hour shift. Obtain the DFTPP spectrum as described in section 11.3.1.

Note: When analyzing samples subject to Wisconsin DNR regulations, a second CCV must be analyzed when second order (quadratic) calibrations are used. One will be analyzed at the lower end of the calibration range and one at a point where the curve can no longer be characterized as first order.

11.4.3. The retention time may be updated for all analytes and surrogates using the CCV standard to account for minor fluctuations or after routine system maintenance.

11.4.4. System Performance Check Compounds (SPCCs): For each daily calibration, a system performance check must be made. For each SPCC compound in the daily calibration standard, a minimum response factor of 0.050 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.5. Calibration Check Compounds (CCCs): After the system performance check, CCCs listed in Section 11.3.6 are used to check the validity of the initial calibration.

Calculate the percent drift using:

$$\%\ Drift = \frac{C_t - C_m}{C_t} \times 100$$

where:
C_t = Calibration Check Compound standard concentration.
C_m = Measured concentration using selected quantitation method.

If the percent drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins. If the % RSD for non-CCC compounds exceeds 30%, the analyst must determine if the response is sufficient to attain MRL for that analyte and any hits for that analyte must be rerun for quantitation.

Note: DoD acceptance criteria is ± 20% from true value for all target compounds and surrogates in the CCV.

11.4.6. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.4.7. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.5. GC/MS Analysis

11.5.1. Evaluate FID screen and make proper dilution (See FID screening SOP).

11.5.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.5.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.5.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extracts in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. Refer to Section 8.0 of Method 8270C for general QC protocol. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270C Section 8.3 for this requirement and acceptance criteria.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *for The Determination of Method Detection Limits and Limits of Detection.*

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

Note: DoD requires no analytes detected at > ½ the RL or 1/10 the regulatory limit, which ever is grater. For common laboratory contaminates there should be no detection > the RL.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix

spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Attachment A. The accuracy of the analysis is controlled on a subset of target analytes. If the project analyte list is fewer than 20 analytes, all are considered control analytes. Analytes which are used for control analytes are listed in Table 6. For DoD projects all project target analytes are considered control analytes. If the LCS recovery for any control analyte fails acceptance limits, corrective action is required. If instrument corrective action is not applicable or ineffective, re-extraction of the associated samples is required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated samples. Project-specific requirements may require all compounds to be treated as control analytes, or dictate use of project acceptance criteria.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = recovered concentration in the MS
R2 = recovered concentration in the DMS

The acceptance limits for the MS/DMS recovery are given in Attachment A. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. The RPD acceptance limits are 30% for water and 40% for soils, sediments, and solids. Project-specific requirements may dictate the use of project acceptance criteria.

12.3.4. The acceptance limits for the surrogates are given in Attachment A. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

13.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks appear to represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.2. For samples containing components not associated with the calibration standards, a library search may be made of the purpose of tentative identification. Refer to method 8270C for guidance on tentatively identified compound (TIC) identification and quantification.

13.3. Quantitation and Calculations

13.3.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions.

When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

Where	Cex	=	Concentration in extract in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.4. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

13.5. Reporting

13.5.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file that the Stealth reporting system uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5.2. As an alternative, reports are generated using Excel© templates located in R:\SVM\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.5.3. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14. CONTINGENCIES FOR HANDLING OUT-OF- CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *the Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses Methylene Chloride and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

17.3. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 3 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP *for Documentation of Technical Personnel Training*.

18.2.1. NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8270C, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

19.2. 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf

19.3. *DoD Quality Systems Manual for Environmental Laboratories* Version 4.1 4/22/09.

CHANGES SINCE THE LAST REVISION

19.4. Sec 11.2 Removed reference to 8000C and SC.
19.5. Sec 11.3.9 Added DoD calibration requirement not allowing use of grand mean in calibrations.
19.6. Sec 11.3.13 Added DoD ICV acceptance criteria.
19.7. Sec 11.4.5 Added DoD CCV acceptance criteria.
19.8. Sec 12.3 Added DoD MB requirements.
19.9. Sec 18 Added training requirements.
19.10. Sec 19 Added DoD reference.
19.11. Sec 11.3.5 Added retention window positioning steps during ICAL.
19.12. Sec 11.4.3 Added retention window positioning step using CCV.

TABLE 1

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
N-Nitrosodimethylamine	25	2.0
Aniline	25	1.0
Bis(2-chloroethyl) Ether	10	0.33
1,2-Dichlorobenzene	10	0.33
1,3-Dichlorobenzene	10	0.33
1,4-Dichlorobenzene	10	0.33
Bis(2-chloroisopropyl) Ether	10	0.33
N-Nitrosodi-n-propylamine	10	0.33
Hexachloroethane	10	0.33
Nitrobenzene	10	0.33
Isophorone	10	0.33
Bis(2-chloroethoxy)methane	10	0.33
1,2,4-Trichlorobenzene	10	0.33
Naphthalene	10	0.33
4-Chloroaniline	10	0.33
Hexachlorobutadiene	10	0.33
2-Methylnaphthalene	10	0.33
Hexachlorocyclopentadiene	10	0.33
2-Chloronaphthalene	10	0.33
2-Nitroaniline	25	2.0
Dimethyl Phthalate	10	0.33
Acenaphthylene	10	0.33
3-Nitroaniline	25	2.0
Acenaphthene	10	0.33
Dibenzofuran	10	0.33
2,4-Dinitrotoluene	10	0.33
2,6-Dinitrotoluene	10	0.33
Diethyl Phthalate	10	0.33

TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
4-Chlorophenyl Phenyl Ether	10	0.33
Fluorene	10	0.33
4-Nitroaniline	25	2.0
N-Nitrosodiphenylamine	10	0.33
4-Bromophenyl Phenyl Ether	10	0.33
Hexachlorobenzene	10	0.33
Phenanthrene	10	0.33
Anthracene	10	0.33
Di-n-butyl Phthalate	10	0.33
Fluoranthene	10	0.33
Pyrene	10	0.33
Butylbenzyl Phthalate	10	0.33
3,3'-Dichlorobenzidine	25	2.0
Benz(a)anthracene	10	0.33
Bis(2-ethylhexyl) Phthalate	10	0.33
Chrysene	10	0.33
Di-n-octyl Phthalate	10	0.33
Benzo(b)fluoranthene	10	0.33
Benzo(k)fluoranthene	10	0.33
Benzo(a)pyrene	10	0.33
Indeno(1,2,3-c,d)pyrene	10	0.33
Dibenz(a,h)anthracene	10	0.33
Benzo(g,h,i)perylene	10	0.33
Phenol	10	0.33
2-Chlorophenol	10	0.33
Benzyl Alcohol	10	0.33
2-Methylphenol	10	0.33
3- and 4-Methylphenol (coeluting cpds)	10	0.33

TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
2-Nitrophenol	10	0.33
2,4-Dimethylphenol	10	0.33
Benzoic Acid	25	2.0
2,4-Dichlorophenol	10	0.33
4-Chloro-3-methylphenol	10	0.33
2,4,6-Trichlorophenol	10	0.33
2,4,5-Trichlorophenol	10	0.33
2,4-Dinitrophenol	25	2.0
4-Nitrophenol	25	2.0
2-Methyl-4,6-dinitrophenol	25	2.0
Pentachlorophenol	25	2.0

TABLE 1A

SEMI-VOLATILE ORGANIC COMPOUNDS - ADDITIONAL ANALYTES	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
Pyridine	25	0.33
2-Picoline	10	0.60
N-Nitrosodiethylamine	10	0.33
Methyl Methanesulfonate	10	0.33
N-Nitrosoethylmethylamine	10	0.33
Pentachloroethane	10	1.0
Acetophenone	10	0.33
N-Nitrosopyrrolidine	10	0.33
N-Nitrosomorpholine	10	0.33
N-Nitrosopiperidine	10	0.33
O,O,O-Triethyl Phosphorothioate	10	0.33
2,6-Dichlorophenol	10	0.33
Hexachloropropene	10	0.33
N-Nitrosodi-*n*-butylamine	10	0.33
p-Phenylenediamine	10	0.33
Safrole	10	0.33
1,2,4,5-Tetrachlorobenzene	10	0.33
Isosafrole	10	2.0
1,3-Dinitrobenzene	10	0.33
Pentachlorobenzene	10	0.33
1-Naphthylamine	10	0.33
2-Naphthylamine	10	0.33
2,3,4,6-Tetrachlorophenol	10	0.33
Diphenylamine	10	0.33
1,3,5-Trinitrobenzene	25	2.0
Phenacetin	50	2.0
4-Aminobiphenyl	10	0.33
4-Nitroquinoline N-Oxide	10	2.0

TABLE 1A continued

SEMI-VOLATILE ORGANIC COMPOUNDS - ADDITIONAL ANALYTES	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
Total Aramite	50	3.0
3,3'-Dimethylbenzidine	25	2.0
7,12-Dimethylbenz(a)anthracene	10	0.33
Hexachlorophene	150	5.0
3-Methylcholanthrene	10	0.33
N,N-Dimethyl-1-phenethylamine	10	20
2-Acetylaminofluorene	10	0.33
o-Toluidine	10	0.33
Ethyl Methanesulfonate	10	0.33
1,4-Naphthoquinone	10	0.33
5-Nitro-*o*-toluidine	10	0.33
p-Dimethylaminoazobenzene	10	0.33
Pentachloronitrobenzene	50	2.0
Methapyrilene	100	4.0
Chlorobenzilate	10	0.33
2-sec-Butyl-4,6-Dinitrophenol (Dinoseb)	25	1.0
Diallate	10	0.33
Dimethoate	10	0.33
Disulfoton	10	0.33
Famphur	10	0.33
Isodrin	10	0.33
Kepone	100	4.0
Methyl Parathion	10	0.33
Parathion	10	0.33
Phorate	10	0.33
Pronamide	10	0.33
Thionazine	25	2.0

TABLE 2
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

Alternate tuning criteria (from Method 525.2 or CLP OLM03.1) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

TABLE 3
8270 STANDARDS

CALIBRATION
Recommended: Supelco catalog #(or equivalent from other vendors*):

Equity CLP SVM Mix, 1000 ppm
Equity Cal Mix 4, 2000 ppm
Equity N-Nitrosodiphenylamine, 5000ppm
Equity Benzidines mix, 2000ppm

Accustandard 8270 surrogate mix, 4000ppm

Prepare 1 ml of each calibration point from a new unopened ampule.
Calibration curve: 5 ppm, 10 ppm, 20 ppm, 50 ppm, 80 ppm, 120 ppm, 160 ppm, and 200 ppm.
Add 10ul internal standard when curve is prepared.
Place ~ 100 µl in autosample vial insert, run, and discard after verifying data is acceptable.
Store in 5 ml amber mininert vials (2-5 ml vials for 10 ml) at -10°C. Expiration is 1 year from date prepared.
Order more solutions when down to one unopened ampule.

ICV
The ICV standard is prepared from the following Accustandard stock standards or equivalent from other vendors*:
Accustandard CLP-HC-BNR (2000ppm), CLP-HC-AR (2000ppm), Z-014ER3 (2000ppm), Z-014F (2000ppm), M-8270-SS (4000ppm), and App-9-114-10x (1000ppm).
Add 10 µl internal standard (Z-014J) for each 1 ml of ICV prepared.
Place in autosampler vial, analyze, recap, and refrigerate.
Expiration is 1 year after ICV was prepared or the expiration date of the stock standard whichever is earliest.

CCV & TUNE
Use the same solutions that were used for the calibration curve and the following:

AccuStandard (recommended) catalog #(or equivalent from other vendors*):

M-625C-3-1000X 1 ml 25 mg/ml DFTPP

Prepare 1 ml of 50 ppm 8270 CCV standard, place in autosample vial and cap with red crimp top. 50ppm is the nominal concentration.
Expiration date is 1 week after CCV was prepared.

RECAP AND STORE IMMEDIATELY AFTER INJECTING
Store remaining stock solutions in 1 ml amber mininert vial. Expiration date is six months after ampule is opened. Order when down to one unopened ampule.

* Vendor must be A2LA and/or ISO9000 certified.

TABLE 4
QC Standards

Supelco Parent	Initial Concentration	Dilution (mixed)*	Final Conc.
8270 Surrogate			
B/N Surrogate Mix (cat no. 86-1377)	5000 ug/mL	20mL to 1000mL in MeOH	100 ug/mL
Acid Surrogate Mix (cat no 86-1376)	10000 ug/mL	15mL to 1000mL in MeOH	150 ug/mL
8270 Matrix Spike (mixed solution)			
CLP Semivolatiles Mix (cat no. 5S-06508)	1000 ug/mL	10mL to 100mL in MeOH	100 ug/mL
Benzidines Mix (cat no. 48467)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
N-Nitroso-diphenylamine (cat no. 46702-U)	5000 ug/mL	2mL to 100mL in MeOH	100 ug/mL
Misc mix (cat no. 86-1148)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
2,3,4,5-Tetrachlorophenol (cat no. 79131)	1000 ug/mL	10mL to 100mL in MeOH	100 ug/mL
1-Methylnaphthalene (cat no. 48162)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
Appendix 9 Mix (cat. no. App-9-186-20x)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL

Each time the QC standards are prepared, the supervisor should be made aware so that re-ordering can occur to maintain an adequate backup supply.

* For surrogate solution, split the total volume made into 4 bottles for storage and use. To avoid waste, the quantity made can be varied as anticipated for workload. For spike solution, split the total volume into 2 bottles for storage and use.

Standards Expiration: Unopened = 6 months from preparation date. Opened = 3 months from opening, or original 6 months, whichever is first. When breaking the seal on an unopened bottle, write the new expiration date on the bottle and in the standards logbook, with initial and date. Absolutely no expired standards are to be kept in the refrigerator.

TABLE 5
SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d4 Internal Standard		
N-Nitrosodimethylamine	1,2-Dichlorobenzene	2-Methylphenol
Aniline	1,3-Dichlorobenzene	3- and 4-Methylphenol (coeluting cpds)
2-Fluorophenol (surrogate)	1,4-Dichlorobenzene	2-Picoline
Bis(2-chloroethyl) Ether	N-Nitrosodi-n-propylamine	Bis(2-chloroisopropyl) Ether
Phenol-d5 (surrogate)	Hexachloroethane	N-Nitrosopyrrolidine
Phenol	Methyl Methanesulfonate	N-Nitrosomorpholine
2-Chloropheol	N-Nitrosoethylmethylamine	*o*-Toluidine
Benzyl Alcohol	Acetophenone	Ethyl Methanesulfonate
Pyridine	N-Nitrosodiethylamine	Pentachloroethane
Naphthalene-d8 Internal Standard		
Nitrobenzene-d5(surrogate)	Hexachlorobutadiene	N-Nitrosodi-*n*-butylamine
Nitrobenzene	2-Methylnaphthalene	N-Nitrosopiperidine
Isophorone	2-Nitrophenol	N,N-Dimethyl-1-phenethylamine
Bis(2-chloroethoxy)methane	2,4-Dimethylphenol	O,O,O-Triethyl Phosphorothioate
1,2,4-Trichlorobenzene	Benzoic Acid	Hexachloropropene
Naphthalene	2,4-Dichlorophenol	*p*-Phenylenediamine
4-Chloroaniline	4-Chloro-3-methylphenol	Safrole
	2,6-Dichlorophenol	1,2,4,5-Tetrachlorobenzene
Acenaphthene-d10 Internal Standard		
2-Fluorobiphenyl (surrogate)	2,4-Dinitrotoluene	1-Naphthylamine
Hexachlorocyclopentadiene	2,6-Dinitrotoluene	2-Naphthylamine
2-Chloronaphthalene	Diethyl Phthalate	2,3,4,6-Tetrachlorophenol
2-Nitroaniline	4-Chlorophenyl Phenyl Ether	2,4,6-Tribromophenol (surrogate)
3-Nitroaniline	Fluorene	Pentachlorobenzene
4-Nitroaniline	4-Nitrophenol	1,3-Dinitrobenzene
Dimethyl Phthalate	2,4,6-Trichlorophenol	1,4-Naphthoquinone
Acenaphthylene	2,4,5-Trichlorophenol	5-Nitro-*o*-toluidine
Acenaphthene	2,4-Dinitrophenol	Thionazine
Dibenzofuran	Isosafrole	Diphenylamine

TABLE 5 continued

Pnenanthrene-d10 Internal Standard		
N-Nitrosodiphenylamine	Pentachlorophenol	Pentachloronitrobenzene
4-Bromophenyl Phenyl Ether	1,3,5-Trinitrobenzene	Disulfoton
Hexachlorobenzene	Phorate	2-sec-Butyl-4,6-Dinitrophenol (Dinoseb)
Phenanthrene	Phenacetin	Methyl Parathion
Anthracene	Diallate	4-Nitroquinoline N-Oxide
Di-n-butyl Phthalate	Dimethoate	Parathion
Fluoranthene	4-Aminobiphenyl	Methapyrilene
2-Methyl-4,6-dinitrophenol	Pronamide	Isodrin
Chrysene-d12 Internal Standard		
Pyrene	Bis(2-ethylhexyl) Phthalate	Chlorobenzilate
Butylbenzyl Phthalate	Chrysene	Kepone
Benzidine	Terphenyl-d14 (surrogate)	3,3'-Dimethylbenzidine
3,3'-Dichlorobenzidine	Total Aramite	Famphur
Benz(a)anthracene	*p*-Dimethylaminoazobenzene	2-Acetylaminofluorene
Perylene-d12 Internal Standard		
Di-n-octyl Phthalate	Indeno(1,2,3-c,d)pyrene	Hexachlorophene
Benzo(b)fluoranthene	Dibenz(a,h)anthracene	3-Methylcholanthrene
Benzo(k)fluoranthene	Benzo(g,h,i)perylene	
Benzo(a)pyrene	7,12-Dimethylbenz(a)anthracene	

TABLE 6
Control Analytes for Non-DoD Projects

1,2,4-Trichlorobenzene
1,4-Dichlorobenzene
2,4-Dinitrotoluene
2-Chloronaphthalene
2-Chlorophenol
4-Bromophenyl Phenyl Ether
4-Chloro-3-methylphenol
4-Nitrophenol
Acenaphthene
Benzo(a)pyrene
Diethyl Phthalate
Hexachloroethane
N-Nitrosodi-n-propylamine
Pentachlorophenol
Phenol
Pyrene

ATTACHMENT A

Method Reporting Limits
Method Detection Limits
QC Acceptance Criteria

Method Information

Method: 8270C
Prep: EPA 3541
Matrix: SOIL
Title: Base Neutral/Acid Semivolatile Organic Compounds

Source: SW-846
Revision: 7/8/2009

Method ID: MJ250

Optimal Prep
Sample Amount: 30.00 g
Final Volume: 1.00 ml

Hold Time
Analytical : 40 days from date Prep
Preparation : 14 days from date Sampled

Analyte Name	*Parm Type*	*Type*	*MDL*	*MRL*	*Surr/MS Limits*	*DMS RPD*	*LCS Limits*	*DLCS RPD*	*Units*	*Analyte ID*
1,4-Dichlorobenzene-d4	INT_STD				.5-2		-		mg/Kg	3855-82-1
Acenaphthene-d10	INT_STD				.5-2		-		mg/Kg	15067-26-2
Chrysene-d12	INT_STD				.5-2		-		mg/Kg	1719-03-5
Naphthalene-d8	INT_STD				.5-2		-		mg/Kg	1146-65-2
Perylene-d12	INT_STD				.5-2		-		mg/Kg	1520-96-3
Phenanthrene-d10	INT_STD				.5-2		-		mg/Kg	1517-22-2
1,2,4-Trichlorobenzene	MS		0.0110	0.33	28-105	40	42-91	40	mg/Kg	120-82-1
1,4-Dichlorobenzene	MS	CCC	0.0175	0.33	28-95	40	41-85	40	mg/Kg	106-46-7
2,4-Dinitrotoluene	MS		0.0149	0.33	36-127	40	48-119	40	mg/Kg	121-14-2
2-Chlorophenol	MS		0.0099	0.33	27-96	40	42-86	40	mg/Kg	95-57-8
4-Chloro-3-methylphenol	MS	CCC	0.0166	0.33	13-122	40	44-101	40	mg/Kg	59-50-7
4-Nitrophenol	MS	SPCC	0.146	2.0	12-141	40	41-121	40	mg/Kg	100-02-7
Acenaphthene	MS	CCC	0.0134	0.33	30-113	40	49-97	40	mg/Kg	83-32-9
N,N-Dimethylaniline	MS		0.0604	0.33	70-130	40	70-130	40	mg/Kg	121-69-7
N-Nitrosodi-n-propylamine	MS	SPCC	0.0191	0.33	32-107	40	41-96	40	mg/Kg	621-64-7
Pentachlorophenol	MS	CCC	0.125	2.0	10-135	40	39-112	40	mg/Kg	87-86-5
Phenol	MS	CCC	0.0195	0.33	18-106	40	40-87	40	mg/Kg	108-95-2
Pyrene	MS		0.0140	0.33	24-126	40	45-116	40	mg/Kg	129-00-0

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4,6-Tribromophenol	SURR				20-123		-		PERCENT	118-79-6
2-Fluorobiphenyl	SURR				32-104		-		PERCENT	321-60-8
2-Fluorophenol	SURR				20-83		-		PERCENT	367-12-4
Nitrobenzene-d5	SURR				29-100		-		PERCENT	4165-60-0
Phenol-d6	SURR				23-90		-		PERCENT	13127-88-3
Terphenyl-d14	SURR				37-133		-		PERCENT	1718-51-0
1,2,3,4-Tetrachlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	634-66-2
1,2,3,5-Tetrachlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	634-90-2
1,2,3-Trichlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	87-61-6
1,2,4,5-Tetrachlorobenzene	TIC_TRG		0.020	0.33	70-130	40	70-130	40	mg/Kg	95-94-3
1,3,5-Trichlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	108-70-3
1-Methylnaphthalene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	90-12-0
2,3,7,8-Tetrachlorodibenzo-p-(	TIC_TRG		0.33	0.33	-		-		mg/Kg	1746-01-6
Acrylamide	TIC_TRG		50	50	70-130	40	70-130	40	mg/Kg	79-06-1
Diisobutyl Phthalate	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	84-69-5
Diphenylamine	TIC_TRG		0.010	0.33	70-130	40	70-130	40	mg/Kg	122-39-4
Hexachlorocyclohexane	TIC_TRG		0.67	0.67	-		-		mg/Kg	608-73-1
HPMO	TIC_TRG		0.121	2.0	-		-		mg/Kg	3375-84-6
OPMO	TIC_TRG		0.0902	2.0	-		-		mg/Kg	CASID30031
Pentachlorobenzene	TIC_TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	608-93-5
1,2-Dichlorobenzene	TRG		0.0179	0.33	29-93	40	41-86	40	mg/Kg	95-50-1
1,2-Diphenylhydrazine	TRG		0.0146	0.33	44-107	40	45-111	40	mg/Kg	122-66-7
1,3,5-Trinitrobenzene	TRG		0.068	0.67	70-130	40	70-130	40	mg/Kg	99-35-4
1,3-Dichlorobenzene	TRG		0.0183	0.33	28-91	40	41-84	40	mg/Kg	541-73-1
1,3-Dinitrobenzene	TRG		0.073	0.33	70-130	40	70-130	40	mg/Kg	99-65-0
1,4-Dichlorobutane	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	110-56-5
1,4-Dioxane	TRG		0.13	0.67	70-130	40	70-130	40	mg/Kg	123-91-1
1,4-Naphthoquinone	TRG		0.065	0.33	70-130	40	70-130	40	mg/Kg	130-15-4
1-Chloronaphthalene	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	90-13-1
1-Methyl-2-pyrrolidinone	TRG		0.095	0.33	70-130	40	33-140	40	mg/Kg	872-50-4
1-Naphthylamine	TRG		0.036	0.33	70-130	40	70-130	40	mg/Kg	134-32-7
2,3,4,6-Tetrachlorophenol	TRG		0.046	1.0	45-110	40	45-110	40	mg/Kg	58-90-2
2,4,5-Trichlorophenol	TRG		0.0171	0.33	27-115	40	46-103	40	mg/Kg	95-95-4
2,4,6-Trichlorophenol	TRG	CCC	0.0143	0.33	24-119	40	46-101	40	mg/Kg	88-06-2
2,4-Dichlorophenol	TRG	CCC	0.0164	0.33	16-119	40	43-94	40	mg/Kg	120-83-2

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4-Dimethylphenol	TRG		0.0151	0.33	10-117	40	16-89	40	mg/Kg	105-67-9
2,4-Dinitrophenol	TRG	SPCC	0.112	2.0	10-145	40	28-116	40	mg/Kg	51-28-5
2,6-Dichlorophenol	TRG		0.016	0.33	70-130	40	70-130	40	mg/Kg	87-65-0
2,6-Diisopropylnaphthalene	TRG		0.33	0.33	14-136	40	14-136	40	mg/Kg	24157-81-1
2,6-Dinitrotoluene	TRG		0.0156	0.33	36-125	40	49-114	40	mg/Kg	606-20-2
2-Acetylaminofluorene	TRG		0.027	4.0	70-130	40	70-130	40	mg/Kg	53-96-3
2-Chloronaphthalene	TRG		0.0100	0.33	26-114	40	44-95	40	mg/Kg	91-58-7
2-Methyl-4,6-dinitrophenol	TRG		0.1434	2.0	10-138	40	41-119	40	mg/Kg	534-52-1
2-Methylnaphthalene	TRG		0.0110	0.33	30-105	40	44-95	40	mg/Kg	91-57-6
2-Methylphenol	TRG		0.0167	0.33	14-103	40	35-92	40	mg/Kg	95-48-7
2-Naphthylamine	TRG		0.063	0.33	70-130	40	70-130	40	mg/Kg	91-59-8
2-Nitroaniline	TRG		0.0169	2.0	34-121	40	48-108	40	mg/Kg	88-74-4
2-Nitrophenol	TRG	CCC	0.0139	0.33	20-119	40	44-95	40	mg/Kg	88-75-5
2-Picoline	TRG		0.055	0.67	70-130	40	70-130	40	mg/Kg	109-06-8
3,3'-Dichlorobenzidine	TRG		0.0270	2.0	10-120	40	38-116	40	mg/Kg	91-94-1
3,3'-Dimethylbenzidine	TRG		1.2	2.0	70-130	40	70-130	40	mg/Kg	119-93-7
3-Methylcholanthrene	TRG		0.062	0.33	70-130	40	70-130	40	mg/Kg	56-49-5
3-Nitroaniline	TRG		0.175	2.0	15-123	40	46-111	40	mg/Kg	99-09-2
3-Nitrophenol	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	554-84-7
4-Aminobiphenyl	TRG		0.065	0.33	70-130	40	70-130	40	mg/Kg	92-67-1
4-Bromophenyl Phenyl Ether	TRG		0.0122	0.33	32-127	40	49-110	40	mg/Kg	101-55-3
4-Chloroaniline	TRG		0.0144	0.33	11-98	40	37-96	40	mg/Kg	106-47-8
4-Chlorophenyl Phenyl Ether	TRG		0.0160	0.33	35-115	40	46-105	40	mg/Kg	7005-72-3
4-Methylphenol	TRG		0.0168	0.33	10-111	40	38-94	40	mg/Kg	106-44-5
4-Nitroaniline	TRG		0.179	2.0	10-137	40	39-120	40	mg/Kg	100-01-6
4-Nitroquinoline N-Oxide	TRG		0.068	3.0	70-130	40	70-130	40	mg/Kg	56-57-5
5-Nitro-o-toluidine	TRG		0.011	0.33	70-130	40	70-130	40	mg/Kg	99-55-8
7,12-Dimethylbenz(a)anthrace	TRG		0.015	0.33	70-130	40	70-130	40	mg/Kg	57-97-6
a,a-Dimethylphenethylamine	TRG		0.23	1.0	70-130	40	70-130	40	mg/Kg	122-09-8
Acenaphthylene	TRG		0.016	0.33	18-128	40	46-99	40	mg/Kg	208-96-8
Acetophenone	TRG		0.023	0.33	70-130	40	70-130	40	mg/Kg	98-86-2
Aniline	TRG		0.0216	1.0	10-92	40	24-92	40	mg/Kg	62-53-3
Anthracene	TRG		0.0139	0.33	21-134	40	50-108	40	mg/Kg	120-12-7
Aramite, Total	TRG		0.056	2.0	70-130	40	70-130	40	mg/Kg	140-57-8
Atrazine	TRG		0.017	0.33	70-130	40	70-130	40	mg/Kg	1912-24-9

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Azobenzene	TRG		0.0146	0.33	70-130	40	70-130	40	mg/Kg	103-33-3
Benz(a)anthracene	TRG		0.0123	0.33	33-121	40	54-109	40	mg/Kg	56-55-3
Benzaldehyde	TRG		0.021	0.33	70-130	40	70-130	40	mg/Kg	100-52-7
Benzidine	TRG		0.42	2.0	10-81	40	10-81	40	mg/Kg	92-87-5
Benzo(a)pyrene	TRG	CCC	0.0198	0.33	31-122	40	51-114	40	mg/Kg	50-32-8
Benzo(b)fluoranthene	TRG		0.0172	0.33	33-124	40	53-110	40	mg/Kg	205-99-2
Benzo(g,h,i)perylene	TRG		0.0202	0.33	26-126	40	50-110	40	mg/Kg	191-24-2
Benzo(k)fluoranthene	TRG		0.0194	0.33	36-117	40	52-112	40	mg/Kg	207-08-9
Benzoic Acid	TRG		0.139	2.0	0-176	40	10-104	40	mg/Kg	65-85-0
Benzophenone	TRG		0.33	0.33	15-116	40	15-116	40	mg/Kg	119-61-9
Benzyl Alcohol	TRG		0.0168		25-106	40	35-96	40	mg/Kg	100-51-6
Biphenyl	TRG		0.009	0.33	70-130	40	70-130	40	mg/Kg	92-52-4
Bis(2-chloroethoxy)methane	TRG		0.0110	0.33	33-106	40	45-93	40	mg/Kg	111-91-1
Bis(2-chloroethyl) Ether	TRG		0.0117	0.33	29-103	40	42-88	40	mg/Kg	111-44-4
Bis(2-chloroisopropyl) Ether	TRG		0.0141	0.33	25-106	40	39-94	40	mg/Kg	39638-32-9
Bis(2-ethylhexyl) Phthalate	TRG		0.0186	0.33	35-126	40	51-115	40	mg/Kg	117-81-7
Butyl Benzyl Phthalate	TRG		0.0163	0.33	34-125	40	51-114	40	mg/Kg	85-68-7
Caprolactam	TRG		0.147	0.67	70-130	40	70-130	40	mg/Kg	105-60-2
Carbazole	TRG		0.0112	0.33	49-118	40	51-113	40	mg/Kg	86-74-8
Chlorobenzilate	TRG		0.020	0.33	70-130	40	70-130	40	mg/Kg	510-15-6
Chrysene	TRG		0.0118	0.33	33-121	40	53-109	40	mg/Kg	218-01-9
Diallate	TRG		0.022	0.33	70-130	40	70-130	40	mg/Kg	2303-16-4
Diazinon	TRG		0.33	0.33	22-135	40	22-135	40	mg/Kg	333-41-5
Dibenz(a,h)anthracene	TRG		0.0275	0.33	40-118	40	53-114	40	mg/Kg	53-70-3
Dibenzofuran	TRG		0.0118	0.33	35-112	40	47-101	40	mg/Kg	132-64-9
Dicyclopentadiene	TRG		0.032	0.33	70-130	40	70-130	40	mg/Kg	77-73-6
Diethyl Phthalate	TRG		0.0141	0.33	37-128	40	45-121	40	mg/Kg	84-66-2
Diethylene Glycol Dibenzoate	TRG		0.33	0.33	32-147	40	32-147	40	mg/Kg	120-55-8
Dimethoate	TRG		0.024	0.33	70-130	40	70-130	40	mg/Kg	60-51-5
Dimethyl Phthalate	TRG		0.0164	0.33	36-123	40	48-113	40	mg/Kg	131-11-3
Di-n-butyl Phthalate	TRG		0.0121	0.33	34-139	40	50-124	40	mg/Kg	84-74-2
Di-n-octyl Phthalate	TRG	CCC	0.0240	0.33	36-136	40	51-121	40	mg/Kg	117-84-0
Dinoseb	TRG		0.052	0.33	70-130	40	70-130	40	mg/Kg	88-85-7
Disulfoton	TRG		0.016	0.33	70-130	40	70-130	40	mg/Kg	298-04-4
Ethyl Methanesulfonate	TRG		0.089	0.33	70-130	40	70-130	40	mg/Kg	62-50-0

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Ethylene Glycol Butyl Ether (E	TRG		0.0058	0.33	34-96	40	34-96	40	mg/Kg	111-76-2
Famphur	TRG		0.071	4.0	70-130	40	70-130	40	mg/Kg	52-85-7
Fluoranthene	TRG	CCC	0.0115	0.33	25-135	40	48-116	40	mg/Kg	206-44-0
Fluorene	TRG		0.0130	0.33	31-116	40	46-103	40	mg/Kg	86-73-7
Hexachlorobenzene	TRG		0.0147	0.33	37-119	40	50-109	40	mg/Kg	118-74-1
Hexachlorobutadiene	TRG	CCC	0.0141	0.33	27-107	40	42-91	40	mg/Kg	87-68-3
Hexachlorocyclopentadiene	TRG	SPCC	0.0125	0.33	10-79	40	10-68	40	mg/Kg	77-47-4
Hexachloroethane	TRG		0.0216	0.33	31-91	40	40-87	40	mg/Kg	67-72-1
Hexachlorophene	TRG		1.2	6.7	70-130	40	70-130	40	mg/Kg	70-30-4
Hexachloropropene	TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	1888-71-7
Indeno(1,2,3-cd)pyrene	TRG		0.0389	0.33	29-132	40	53-112	40	mg/Kg	193-39-5
Isodrin	TRG		0.0089	0.33	70-130	40	70-130	40	mg/Kg	465-73-6
Isophorone	TRG		0.0140	0.33	33-103	40	42-93	40	mg/Kg	78-59-1
Isosafrole	TRG		0.017	0.67	70-130	40	70-130	40	mg/Kg	120-58-1
Kepone	TRG		0.39	4.0	70-130	40	70-130	40	mg/Kg	143-50-0
Malathion	TRG		0.83	0.83	70-130	40	70-130	40	mg/Kg	121-75-5
Methapyrilene	TRG		0.091	1.0	70-130	40	70-130	40	mg/Kg	91-80-5
Methyl Methanesulfonate	TRG		0.089	0.33	70-130	40	70-130	40	mg/Kg	66-27-3
Methyl Parathion	TRG		0.013	0.33	70-130	40	70-130	40	mg/Kg	298-00-0
Naphthalene	TRG		0.0144	0.33	31-103	40	44-92	40	mg/Kg	91-20-3
n-Dodecane	TRG		0.33	0.33	26-95	40	26-95	40	mg/Kg	112-40-3
Nitrobenzene	TRG		0.0261	0.33	34-99	40	44-92	40	mg/Kg	98-95-3
N-Nitrosodiethylamine	TRG		0.029	0.33	70-130	40	70-130	40	mg/Kg	55-18-5
N-Nitrosodimethylamine	TRG		0.0251	2.0	20-98	40	34-93	40	mg/Kg	62-75-9
N-Nitrosodi-n-butylamine	TRG		0.11	0.33	70-130	40	70-130	40	mg/Kg	924-16-3
N-Nitrosodiphenylamine	TRG	CCC	0.018	0.33	14-143	40	44-113	40	mg/Kg	86-30-6
N-Nitrosomethylethylamine	TRG		0.099	0.33	70-130	40	70-130	40	mg/Kg	10595-95-6
N-Nitrosomorpholine	TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	59-89-2
N-Nitrosopiperidine	TRG		0.023	0.33	70-130	40	70-130	40	mg/Kg	100-75-4
N-Nitrosopyrrolidine	TRG		0.092	0.33	70-130	40	70-130	40	mg/Kg	930-55-2
O,O,O-Triethyl Phosphorothio	TRG		0.022	0.33	70-130	40	70-130	40	mg/Kg	126-68-1
o-Toluidine	TRG		0.017	0.33	70-130	40	70-130	40	mg/Kg	95-53-4
Parathion	TRG		0.084	0.33	70-130	40	70-130	40	mg/Kg	56-38-2
p-Dimethylaminoazobenzene	TRG		0.012	0.33	70-130	40	70-130	40	mg/Kg	60-11-7
Pentachloroethane	TRG		0.015	1.0	70-130	40	70-130	40	mg/Kg	76-01-7

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Pentachloronitrobenzene	TRG		0.088	2.0	70-130	40	70-130	40	mg/Kg	82-68-8
PGMEA	TRG		0.036	1.0	70-130	40	70-130	40	mg/Kg	108-65-6
Phenacetin	TRG		0.013	2.0	70-130	40	70-130	40	mg/Kg	62-44-2
Phenanthrene	TRG		0.0100	0.33	35-119	40	52-108	40	mg/Kg	85-01-8
Phorate	TRG		0.026	0.33	70-130	40	70-130	40	mg/Kg	298-02-2
Picric Acid	TRG		3.3	3.3	70-130	40	70-130	40	mg/Kg	88-89-1
p-Phenylenediamine	TRG		0.79	2.0	70-130	40	70-130	40	mg/Kg	106-50-3
Pronamide	TRG		0.018	0.33	70-130	40	70-130	40	mg/Kg	23950-58-5
Pyridine	TRG		0.0199	0.33	10-153	40	10-153	40	mg/Kg	110-86-1
Quinoline	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	91-22-5
Safrole	TRG		0.013	0.33	70-130	40	70-130	40	mg/Kg	94-59-7
Sulfotep	TRG		0.018	0.33	70-130	40	70-130	40	mg/Kg	3689-24-5
Thionazin	TRG		0.017	2.0	70-130	40	70-130	40	mg/Kg	297-97-2

Method Information

Method: 8270C
Prep: EPA 3520C
Matrix: WATER
Title: Base Neutral/Acid Semivolatile Organic compounds

Source: SW-846
Revision: 7/8/2009

Method ID: MJ121

Optimal Prep
Sample Amount: 1,000.00 ml
Final Volume: 1.00 ml

Hold Time
Analytical : 40 days from date Prep
Preparation 7 days from date Sampled

Analyte Name	*Parm Type*	*Type*	*MDL*	*MRL*	*Surr/MS Limits*	*DMS RPD*	*LCS Limits*	*DLCS RPD*	*Units*	*Analyte ID*
1,4-Dichlorobenzene-d4	INT_STD				.5-2		-		ug/L	3855-82-1
Acenaphthene-d10	INT_STD				.5-2		-		ug/L	15067-26-2
Chrysene-d12	INT_STD				.5-2		-		ug/L	1719-03-5
Naphthalene-d8	INT_STD				.5-2		-		ug/L	1146-65-2
Perylene-d12	INT_STD				.5-2		-		ug/L	1520-96-3
Phenanthrene-d10	INT_STD				.5-2		-		ug/L	1517-22-2
1,2,4-Trichlorobenzene	MS		0.355	10	49-97	30	51-103	30	ug/L	120-82-1
1,4-Dichlorobenzene	MS	CCC	0.317	10	47-93	30	54-96	30	ug/L	106-46-7
2,4-Dinitrotoluene	MS		0.274	10	62-123	30	61-126	30	ug/L	121-14-2
2-Chlorophenol	MS		0.311	10	49-100	30	59-101	30	ug/L	95-57-8
4-Chloro-3-methylphenol	MS	CCC	0.490	10	61-111	30	65-111	30	ug/L	59-50-7
4-Nitrophenol	MS	SPCC	1.92	25	57-119	30	55-126	30	ug/L	100-02-7
Acenaphthene	MS	CCC	0.281	10	57-107	30	61-110	30	ug/L	83-32-9
N,N-Dimethylaniline	MS		2.22	10	70-130	30	70-130	30	ug/L	121-69-7
N-Nitrosodi-n-propylamine	MS	SPCC	0.496	10	53-109	30	56-115	30	ug/L	621-64-7
Pentachlorophenol	MS	CCC	2.44	25	42-127	30	60-116	30	ug/L	87-86-5
Phenol	MS	CCC	0.324	10	47-101	30	48-105	30	ug/L	108-95-2
Pyrene	MS		0.731	10	52-121	30	53-124	30	ug/L	129-00-0

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4,6-Tribromophenol	SURR				46-127		-		PERCENT	118-79-6
2-Fluorobiphenyl	SURR				48-114		-		PERCENT	321-60-8
2-Fluorophenol	SURR				39-103		-		PERCENT	367-12-4
Nitrobenzene-d5	SURR				46-115		-		PERCENT	4165-60-0
Phenol-d6	SURR				38-107		-		PERCENT	13127-88-3
Terphenyl-d14	SURR				22-146		-		PERCENT	1718-51-0
1-Methylnaphthalene	TIC_TRG		10	10	-		-		ug/L	90-12-0
2,3,7,8-Tetrachlorodibenzo-p-(	TIC_TRG		50	50	-		-		ug/L	1746-01-6
alpha-Terpineol	TIC_TRG		10	10	-		-		ug/L	10482-56-1
Chloroacetaldehyde	TIC_TRG		10	10	-		-		ug/L	107-20-0
HPMO	TIC_TRG		6.15	25	-		-		ug/L	3375-84-6
OPMO	TIC_TRG		3.86	25	-		-		ug/L	CASID30031
Paraldehyde	TIC_TRG		10	10	-		-		ug/L	123-63-7
Toluene diisocyanate	TIC_TRG		10	10	-		-		ug/L	CASID30165
1,2,4,5-Tetrachlorobenzene	TRG		0.26	10	70-130	30	70-130	30	ug/L	95-94-3
1,2-Dichlorobenzene	TRG		0.431	10	47-94	30	55-97	30	ug/L	95-50-1
1,2-Diphenylhydrazine	TRG		0.509	10	54-118	30	58-117	30	ug/L	122-66-7
1,3,5-Trinitrobenzene	TRG		0.38	25	70-130	30	70-130	30	ug/L	99-35-4
1,3-Dichlorobenzene	TRG		0.352	10	45-92	30	55-95	30	ug/L	541-73-1
1,3-Dinitrobenzene	TRG		0.52	10	70-130	30	70-130	30	ug/L	99-65-0
1,4-Dichlorobutane	TRG		1	10	70-130	30	70-130	30	ug/L	110-56-5
1,4-Dioxane	TRG		3.7	25	70-130	30	70-130	30	ug/L	123-91-1
1,4-Naphthoquinone	TRG		0.21	10	70-130	30	70-130	30	ug/L	130-15-4
1-Chloronaphthalene	TRG		1	10	70-130	30	70-130	30	ug/L	90-13-1
1-Methyl-2-pyrrolidinone	TRG		2.2	25	70-130	30	70-130	30	ug/L	872-50-4
1-Naphthylamine	TRG		0.70	10	70-130	30	70-130	30	ug/L	134-32-7
2,3,4,6-Tetrachlorophenol	TRG		0.55	10	56-124	30	56-124	30	ug/L	58-90-2
2,3,5,6-Tetrachlorophenol	TRG		0.62	10	70-130	30	70-130	30	ug/L	935-95-5
2,4,5-Trichlorophenol	TRG		0.381	10	60-112	30	62-117	30	ug/L	95-95-4
2,4,6-Trichlorophenol	TRG	CCC	0.203	10	60-113	30	63-117	30	ug/L	88-06-2
2,4-Dichlorophenol	TRG	CCC	0.297	10	55-105	30	59-107	30	ug/L	120-83-2
2,4-Dimethylphenol	TRG		0.264	10	22-116	30	32-100	30	ug/L	105-67-9
2,4-Dinitrophenol	TRG	SPCC	2.22	25	38-122	30	33-126	30	ug/L	51-28-5
2,6-Dichlorophenol	TRG		0.48	10	70-130	30	70-130	30	ug/L	87-65-0
2,6-Dinitrotoluene	TRG		0.349	10	69-113	30	65-122	30	ug/L	606-20-2

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2-Acetylaminofluorene	TRG		0.23	100	70-130	30	70-130	30	ug/L	53-96-3
2-Chloronaphthalene	TRG		0.290	10	48-106	30	54-107	30	ug/L	91-58-7
2-Methyl-4,6-dinitrophenol	TRG		2.12	25	57-120	30	51-127	30	ug/L	534-52-1
2-Methylnaphthalene	TRG		0.239	10	57-100	30	57-106	30	ug/L	91-57-6
2-Methylphenol	TRG		0.328	10	52-100	30	51-108	30	ug/L	95-48-7
2-Naphthylamine	TRG		1.0	10	70-130	30	70-130	30	ug/L	91-59-8
2-Nitroaniline	TRG		0.336	25	23-138	30	62-117	30	ug/L	88-74-4
2-Nitrophenol	TRG	CCC	0.373	10	54-104	30	58-108	30	ug/L	88-75-5
2-Picoline	TRG		4.5	25	70-130	30	70-130	30	ug/L	109-06-8
3- and 4-Methylphenol Coeluti	TRG		0.478	10	-		-		ug/L	CASID30030
3,3'-Dichlorobenzidine	TRG		0.270	25	12-120	30	52-120	30	ug/L	91-94-1
3,3'-Dimethylbenzidine	TRG		5.4	20	70-130	30	70-130	30	ug/L	119-93-7
3-Methylcholanthrene	TRG		0.31	10	70-130	30	70-130	30	ug/L	56-49-5
3-Nitroaniline	TRG		3.25	25	57-119	30	63-120	30	ug/L	99-09-2
3-Nitrophenol	TRG		1	10	70-130	30	70-130	30	ug/L	554-84-7
4-Aminobiphenyl	TRG		1.4	10	70-130	30	70-130	30	ug/L	92-67-1
4-Bromophenyl Phenyl Ether	TRG		0.274	10	61-114	30	64-118	30	ug/L	101-55-3
4-Chloroaniline	TRG		0.375	10	42-117	30	57-111	30	ug/L	106-47-8
4-Chlorophenyl Phenyl Ether	TRG		0.278	10	56-116	30	59-113	30	ug/L	7005-72-3
4-Methylphenol	TRG		0.478	10	54-108	30	47-118	30	ug/L	106-44-5
4-Nitroaniline	TRG		4.03	25	37-130	30	53-127	30	ug/L	100-01-6
4-Nitroquinoline N-Oxide	TRG		4.6	100	70-130	30	70-130	30	ug/L	56-57-5
5-Nitro-o-toluidine	TRG		1.0	10	70-130	30	70-130	30	ug/L	99-55-8
7,12-Dimethylbenz(a)anthrace	TRG		0.32	10	70-130	30	70-130	30	ug/L	57-97-6
a,a-Dimethylphenethylamine	TRG		6.4	25	70-130	30	70-130	30	ug/L	122-09-8
Acenaphthylene	TRG		0.236	10	59-101	30	58-109	30	ug/L	208-96-8
Acetophenone	TRG		0.60	10	70-130	30	70-130	30	ug/L	98-86-2
Aniline	TRG		0.487	25	23-116	30	35-118	30	ug/L	62-53-3
Anthracene	TRG		0.612	10	59-112	30	62-116	30	ug/L	120-12-7
Aramite, Total	TRG		0.52	50	70-130	30	70-130	30	ug/L	140-57-8
Atrazine	TRG		0.46	10	70-130	30	70-130	30	ug/L	1912-24-9
Azobenzene	TRG		0.509	10	70-130	30	70-130	30	ug/L	103-33-3
Benz(a)anthracene	TRG		0.591	10	63-110	30	69-113	30	ug/L	56-55-3
Benzaldehyde	TRG		0.50	10	70-130	30	70-130	30	ug/L	100-52-7
Benzidine	TRG		19	50	10-181	30	10-181	30	ug/L	92-87-5

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Benzo(a)pyrene	TRG		0.651	10	54-123	30	57-124	30	ug/L	50-32-8
Benzo(b)fluoranthene	TRG		0.584	10	59-117	30	66-117	30	ug/L	205-99-2
Benzo(g,h,i)perylene	TRG		0.812	10	61-116	30	68-116	30	ug/L	191-24-2
Benzo(k)fluoranthene	TRG		0.827	10	57-118	30	63-119	30	ug/L	207-08-9
Benzoic acid	TRG		5.819	25	10-136	30	10-109	30	ug/L	65-85-0
Benzyl alcohol	TRG		0.377	10	51-105	30	53-111	30	ug/L	100-51-6
Biphenyl	TRG		0.66	10	70-130	30	70-130	30	ug/L	92-52-4
Bis(2-chloroethoxy)methane	TRG		0.276	10	55-101	30	57-108	30	ug/L	111-91-1
Bis(2-chloroethyl) Ether	TRG		0.333	10	51-97	30	56-104	30	ug/L	111-44-4
Bis(2-chloroisopropyl) Ether	TRG		0.311	10	43-106	30	53-105	30	ug/L	39638-32-9
Bis(2-ethylhexyl) Phthalate	TRG		1.89	10	58-122	30	64-122	30	ug/L	117-81-7
Butyl Benzyl Phthalate	TRG		0.470	10	60-117	30	64-121	30	ug/L	85-68-7
Caprolactam	TRG		0.58	25	70-130	30	70-130	30	ug/L	105-60-2
Carbazole	TRG		0.237	10	63-122	30	63-122	30	ug/L	86-74-8
Chlorobenzilate	TRG		0.45	10	70-130	30	70-130	30	ug/L	510-15-6
Chrysene	TRG		0.787	10	61-113	30	68-114	30	ug/L	218-01-9
Diallate	TRG		0.47	10	70-130	30	70-130	30	ug/L	2303-16-4
Dibenz(a,h)anthracene	TRG		0.752	10	60-120	30	65-121	30	ug/L	53-70-3
Dibenzofuran	TRG		0.325	10	61-110	30	62-112	30	ug/L	132-64-9
Dicyclopentadiene	TRG		0.59	10	70-130	30	70-130	30	ug/L	77-73-6
Diethyl Phthalate	TRG		0.289	10	61-122	30	62-122	30	ug/L	84-66-2
Dimethoate	TRG		0.69	10	70-130	30	70-130	30	ug/L	60-51-5
Dimethyl Phthalate	TRG		0.254	10	69-110	30	64-119	30	ug/L	131-11-3
Di-n-butyl Phthalate	TRG		0.364	10	61-127	30	65-126	30	ug/L	84-74-2
Di-n-octyl Phthalate	TRG	CCC	0.626	10	57-129	30	60-131	30	ug/L	117-84-0
Dinoseb	TRG		0.42	10	70-130	30	70-130	30	ug/L	88-85-7
Diphenylamine	TRG		0.42	10	70-130	30	70-130	30	ug/L	122-39-4
Disulfoton	TRG		0.57	10	70-130	30	70-130	30	ug/L	298-04-4
Ethyl Methanesulfonate	TRG		0.28	10	70-130	30	70-130	30	ug/L	62-50-0
Famphur	TRG		0.27	10	70-130	30	70-130	30	ug/L	52-85-7
Fluoranthene	TRG	CCC	0.652	10	56-123	30	60-121	30	ug/L	206-44-0
Fluorene	TRG		0.323	10	58-112	30	59-112	30	ug/L	86-73-7
Hexachlorobenzene	TRG		0.628	10	56-115	30	63-117	30	ug/L	118-74-1
Hexachlorobutadiene	TRG	CCC	0.291	10	44-94	30	54-99	30	ug/L	87-68-3
Hexachlorocyclopentadiene	TRG	SPCC	1.21	10	10-58	30	10-60	30	ug/L	77-47-4

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Hexachloroethane	TRG		0.289	10	40-101	30	50-100	30	ug/L	67-72-1
Hexachlorophene	TRG		44	150	70-130	30	70-130	30	ug/L	70-30-4
Hexachloropropene	TRG		0.19	10	70-130	30	70-130	30	ug/L	1888-71-7
Indeno(1,2,3-cd)pyrene	TRG		0.684	10	62-113	30	68-116	30	ug/L	193-39-5
Isodrin	TRG		0.36	10	70-130	30	70-130	30	ug/L	465-73-6
Isophorone	TRG		0.246	10	60-99	30	57-106	30	ug/L	78-59-1
Isosafrole	TRG		0.48	10	70-130	30	70-130	30	ug/L	120-58-1
Kepone	TRG		4.1	20	70-130	30	70-130	30	ug/L	143-50-0
Malathion	TRG		1	25	70-130	30	70-130	30	ug/L	121-75-5
Methapyrilene	TRG		9.3	100	70-130	30	70-130	30	ug/L	91-80-5
Methyl Methanesulfonate	TRG		0.31	10	70-130	30	70-130	30	ug/L	66-27-3
Methyl Parathion	TRG		0.51	10	70-130	30	70-130	30	ug/L	298-00-0
Naphthalene	TRG		0.365	10	51-99	30	51-106	30	ug/L	91-20-3
Nitrobenzene	TRG		0.567	10	31-141	30	56-108	30	ug/L	98-95-3
N-Nitrosodiethylamine	TRG		0.41	10	70-130	30	70-130	30	ug/L	55-18-5
N-Nitrosodimethylamine	TRG		0.479	25	47-100	30	53-109	30	ug/L	62-75-9
N-Nitrosodi-n-butylamine	TRG		0.57	10	70-130	30	64-105	30	ug/L	924-16-3
N-Nitrosodiphenylamine	TRG	CCC	0.48	10	10-135	30	58-121	30	ug/L	86-30-6
N-Nitrosomethylethylamine	TRG		4.6	25	70-130	30	70-130	30	ug/L	10595-95-6
N-Nitrosomorpholine	TRG		0.25	10	70-130	30	70-130	30	ug/L	59-89-2
N-Nitrosopiperidine	TRG		0.32	10	70-130	30	70-130	30	ug/L	100-75-4
N-Nitrosopyrrolidine	TRG		0.39	10	70-130	30	70-130	30	ug/L	930-55-2
O,O,O-Triethyl Phosphorothio	TRG		0.37	10	70-130	30	70-130	30	ug/L	126-68-1
o-Toluidine	TRG		1.4	10	70-130	30	70-130	30	ug/L	95-53-4
Parathion	TRG		0.51	10	70-130	30	70-130	30	ug/L	56-38-2
p-Dimethylaminoazobenzene	TRG		0.31	10	70-130	30	70-130	30	ug/L	60-11-7
Pentachlorobenzene	TRG		0.31	10	70-130	30	70-130	30	ug/L	608-93-5
Pentachloroethane	TRG		0.28	25	70-130	30	70-130	30	ug/L	76-01-7
Pentachloronitrobenzene	TRG		0.23	50	70-130	30	70-130	30	ug/L	82-68-8
PGMEA	TRG		0.96	10	70-130	30	70-130	30	ug/L	108-65-6
Phenacetin	TRG		0.42	50	70-130	30	70-130	30	ug/L	62-44-2
Phenanthrene	TRG		0.482	10	61-111	30	64-116	30	ug/L	85-01-8
Phorate	TRG		0.36	10	70-130	30	70-130	30	ug/L	298-02-2
p-Phenylenediamine	TRG		19	100	70-130	30	70-130	30	ug/L	106-50-3
Pronamide	TRG		0.41	10	70-130	30	70-130	30	ug/L	23950-58-5

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Pyridine	TRG		7.50	25	10-142	30	10-142	30	ug/L	110-86-1
Quinoline	TRG		10	10	70-130	30	70-130	30	ug/L	91-22-5
Safrole	TRG		0.36	10	70-130	30	70-130	30	ug/L	94-59-7
Sulfotep	TRG		0.26	10	70-130	30	70-130	30	ug/L	3689-24-5
Thionazin	TRG		0.71	25	70-130	30	70-130	30	ug/L	297-97-2

TARGET SHEET

SITE NAME: SAN JACINTO RIVER WASTE PITS

CERCLIS I.D.: TXN000606611

TITLE OF DOC.: DRAFT SAMPLING AND ANYLSIS PLAN: SOIL STUDY

DATE OF DOC.: 12/01/2010

NO. OF PGS. THIS TARGET SHEET REPLACES: 70

SDMS #: 9546106 **RELATED #:** 9184235

SENSITIVE ?	X	**MISSING PAGES ?**	
ALTERN. MEDIA ?		**CROSS REFERENCE ?**	

LAB DOCUMENT ?		**LAB NAME:**	
ASC./BOX #:			
CASE #:		**SDG #:**	

COMMENTS : PAGES 562-631 WERE REDACTED FROM THIS DOCUMENT DUE TO FOIA EXEMPTION 4 - CONFIDENTIAL BUSINESS INFORMATION

Columbia Analytical Services™

SOP CHANGE FORM

SOP Title: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Isotope Dilution HRGC/HRMS
SOP Code: HMS-1668A
SOP Revision No.: 4
SOP Date: 08/13/20 (effective 08/20/10)
SOP Section(s) Affected by Change: 11.2.2.1-11.2.2.3, 11.2.3.2, 11.2.4.1-11.2.4.3

Description of Change:
Spiking will now occur prior to mixing solid and tissue samples with sodium sulfate.

11.2.2.1 Add 10-20g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.2.2 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet.

11.2.2.3 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

11.2.3.2 Rather than adding sample/spiking solution to a clean thimble, instead add sample to a clean beaker to record weight. Add 10-20g sodium sulfate and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained. Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3). Record the standard addition on the bench sheet. Transfer sample to ASE cell. Top with sodium sulfate.

11.2.4.1 Add 20-30g anhydrous sodium sulfate to each thimble containing the sample and mix thoroughly to evenly distribute the sodium sulfate. If the mixture is not free-flowing, continue to mix in sodium sulfate until a free-flowing consistency can be obtained.

11.2.4.2 Spike 1.0mL of the Labeled standard spiking solution at 10-20ng/mL (Table 3) into each sample and each QC aliquot. Record this addition on the bench sheet. If lipid determination is to be performed on a sample,

spike 2.0mL of the Labeled standard spiking solution, instead. 11.2.4.3 Spike 1.0mL of the Matrix standard spiking solution at 5ng/mL (Table 3) into the LCS/DLCS aliquots. Record this addition on the bench sheet. These will serve as both the precision and accuracy for the batch.

Reason(s) for Change(s): After some recent investigation, the EPA ORCR released a memo recommending that the recently updated language in some EPA 3500 series methods regarding standard addition should not be followed, and that addition of labeled standards to solid samples should occur following mixture with sodium sulfate to minimize potential low percent recovery issues.

Change(s) Submitted by: Andrew Biddle	Date: 08/17/10

Approvals:

Technical Reviewer Signature: Virginia Loring	Date: 8/17/10
QA PM Signature:	Date: 08/17/10
Department Supervisor/Manager Signature:	Date: 8/17/10

Change(s) Effective Date: 08/17/10

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

TARGET SHEET

SITE NAME: SAN JACINTO RIVER WASTE PITS

CERCLIS I.D.: TXN000606611

TITLE OF DOC.: DRAFT SAMPLING AND ANYLSIS PLAN: SOIL STUDY

DATE OF DOC.: 12/01/2010

NO. OF PGS. THIS TARGET SHEET REPLACES: 96

SDMS #: 9546106 **RELATED #:** 9184235

SENSITIVE ?	X	**MISSING PAGES ?**	
ALTERN. MEDIA ?		**CROSS REFERENCE ?**	

LAB DOCUMENT ?		**LAB NAME:**	
ASC./BOX #:			
CASE #:		**SDG #:**	

COMMENTS : PAGES 634-729 WERE REDACTED FROM THIS DOCUMENT DUE TO FOIA EXEMPTION 4 - CONFIDENTIAL BUSINESS INFORMATION

APPENDIX C
UNMIXING ANALYSIS AND SOURCE IDENTIFICATION

INTRODUCTION

To address uncertainties regarding chemical sources at sites known or suspected to be affected by more than one source, Integral applies unmixing analysis, a numerical technique that provides a robust quantification of potential contributions of each chemical source in each environmental sample. This mathematical technique generates quantitative lines of evidence for characterizing source types within a mixture, and for estimating the relative contribution of each source type to samples collected from a site. As noted in Section 6.1.5 of the RI/FS Work Plan, San Jacinto River Waste Pits (Anchor and Integral 2010), and in Section 1.10.1.2 of the Sampling and Analysis Plan, Sediment Study (Integral and Anchor QEA 2010), unmixing analysis will be used to evaluate the proportional contribution of chemicals in waste materials from the impoundments to the surrounding sediments. Similarly and for the same purpose, unmixing methods will be applied to analysis of soil data.

Initially developed as a technique to help unscramble mixed radio signals, unmixing analysis is still widely used in applications with chromatograms and spectrographic data and digital image analysis. In the past decade, the technique has been used in fields ranging from investigations of ingredient mixtures in food products and fuel blends to face recognition and text mining. Application of unmixing analysis has been increasing in the environmental forensics field because it provides a means to discriminate and quantify the nature and contributions of multiple sources to a site using standard environmental chemistry data sets.

From a set of samples containing complex mixtures of multiple chemicals, unmixing analysis produces quantitative information that can be used to determine the number and characteristics of different source types influencing those samples. When sources have demonstrably characteristic chemical patterns, unmixing analysis generates "end members" that each describe a distinctive source type within the mixture, and uses the framework created by the set of end members to identify the relative contributions of different source types, including background conditions, to each sample.

AVAILABLE METHODS AND APPLICATIONS

Unmixing analysis can resolve data sets that are the result of any mixing physical process where conservation of mass principles apply, including present and historical complete mixing, periodic mixing, active sediment transport, periodic deposition, and discontinuous discharges. The method is not dependent on any specifics regarding mixing processes or timing. The mechanism of mixing—whether a mixture was created all at once in a single event, or by gradual contributions from multiple sources over a period of time—has no influence on the method's ability to distinguish the different mixing end members. Unmixing methods do not produce any information about the history of source contributions or mixing events. If the timing of source releases is known, however, and available information suggests that some contaminant degradation might have occurred, that information can be incorporated into the analysis.

Additional data preparation steps are required if unmixing is applied to data sets influenced by non-conservative processes (such as degradation). These preliminary steps are designed to account for chemical or biological processes, which result in the degradation of source material and/or creation of chemicals not present in the original sources. Degradation or other non-conservative processes can thereby be accounted for before the unmixing method is applied to the data.

Among the many available unmixing algorithms, the most commonly cited in the literature are non-negative (or positive) matrix factorization (NMF or PMF), polytopic vector analysis (PVA), alternating least squares (ALS), and principal components or factor analysis with non-negativity constraints (PCA- or FA-NNC). All unmixing analysis variants implicitly rely on two assumptions: 1) that all samples in the data set were created by the mixing of the same sources in various non-negative proportions (i.e., ≥ 0), and 2) that mass of the chemicals used in the unmixing analysis is conserved. In our own implementation, one explicit assumption that can (optionally) be imposed, if warranted and supported by other lines of evidence, is that the original sources are physically represented by samples within the data set.

Application of unmixing to environmental data sets is growing. The U.S. Environmental Protection Agency (USEPA) has applied two methods of unmixing analysis: PMF and UNMIX. A final report of a USEPA workshop, including case studies and applications of

these two methods is available on the USEPA website[1]. USEPA's most prominent use of unmixing was presented by the USEPA Office of Air Quality Planning and Standards as part of their particulate matter air quality standards assessment (USEPA 2008[2]; within the document titled *Technical Support for State and Tribal Air Quality 24-Hour Fine Particle (PM2.5) Designations*, for example, §4.10.1 Region 10 Nonattainment Areas-Alaska[3]). In addition, the BC Ministry of Environment, Lands and Parks (Canada) used unmixing (PVA) in a technical document in support of the Georgia Basin Ecosystem Initiative (Bright 2001). Unmixing was used to investigate sources of dioxins and mercury in municipal wastewater and treatment biosolids in this study.

Publications in the peer-reviewed scientific literature referencing unmixing methods for environmental data sets have been increasing over time (Table 1). Recent publications (Mas et al. 2010; Mostert et al. 2010) highlight unmixing analysis as a powerful, underutilized technique for applications in environmental forensics.

Table 1. Numbers of peer-reviewed publications referencing the use of unmixing methods for environmental data sets by year.

Publication Year	Record Count	% of 790	Bar Chart
2009	107	13.5 %	
2008	84	10.6 %	
2007	72	9.1 %	
2006	69	8.7 %	
2004	59	7.5 %	

Search Topic=((sediment* or soil* or water* or air) and ((positive or non-negative or nonnegative) and matrix factorization or polytopic vector or unmix*))

Integral has applied unmixing analysis to sites contaminated with dioxins, furans, and polychlorinated biphenyls (PCBs). In one application, unmixing analysis provided a quantitative line of evidence to characterize potential impacts of dioxins and furans from a former industrial facility to soils in neighboring residential yards in an urban-industrial

[1] http://www.epa.gov/ttnamti1/unmixmtg.html
[2] http://www.epa.gove/pmdesignations/2006standards/tech.htm
[3] http://www.epa.gov/pmdesignations/2006standards/final/TSD/tsd_4.0_4.10_4.10.1_r10_AK.pdf

area that had also been influenced by common regional atmospheric sources of dioxins and furans. At another site, unmixing analysis was used to estimate the relative contributions of two different shoreline sources of PCBs to marine sediments within a cleanup site. In addition, unmixing has been applied at the following contaminated sites, each of which provides an example for which the use of unmixing has been documented on a website or in a report.

Rayonier Mill, Port Angeles, Washington—Unmixing (PMF, PVA, ALS) has been used to identify potential sources of dioxins and furans in soils and sediments. The work has been carried out for the Washington State Department of Ecology by a contractor[4].
Lower Duwamish Waterway (T117 EAA), Washington—Unmixing (NMF and ALS) has been used to identify potential sources of dioxins and furans in soil and sediment[5].
Fox River, Wisconsin—Unmixing (FA-NNC) was used to identify sources of PCBs to sediments as well as construct a degradation (dechlorination) model (Imamoglu et al. 2004).
Canadian Soils Study—Unmixing (PVA) was used to study sources of dioxins, furans, and PCBs in soils of abandoned military sites throughout Northern Canada (Grundy et al. 1997)
Newark Bay, New Jersey—Sources of dioxins and their relative contributions sediment in Newark Bay were investigated using PVA (Ehlrich et al. 1994).
Diamond Alkali Superfund Site (Passaic River, New Jersey)—A modified PVA algorithm was employed to construct a model of dioxin degradation in sediments aimed at identifying compositional changes in the original source patterns caused by weathering and/or biological activity (Barabas et al. 2004).
Milwaukee Harbor Estuary, Wisconsin—Sources of PCBs to sediments were quantified using unmixing analysis (PCA-NNC) of sediment core samples (Rachdawong and Christensen 1997).
Philadelphia, Pennsylvania–Camden, New Jersey, metro area—Source identification of atmospheric PCBs and deposition loadings were calculated using PMF and shown to exceed the entire TMDL in the tidal Delaware River (Du and Rodenburg 2007).

[4] http://www.ecy.wa.gov/programs/tcp/sites/rayonierOffProp/rayonierOffPr_hp.htm -- see the July 9 technical workshop presentation
[5] Appendix M - Dioxin Technical Workgroup Findings; http://www.t117.com/docs_technical.aspx

Little Menomonee River, Wisconsin—A source apportionment study for PAHs in sediment was conducted using PMF (Stout & Graan 2010).

Dalian, China—Source identification and contributions of dioxins and PCBs in pine needles near an industrialized coastal city in China were investigated using two unmixing methods (PMF and FA-NNC) in support of the implementation of the Stockholm Convention in China (beginning 2004) (Tian et al. 2008).

SUMMARY

Unmixing analysis provides a powerful numerical technique to generate quantitative lines of evidence that allow analysts to identify source types within samples that are characterized by chemical mixtures. It will be used to evaluate proportional contributions from different sources to sediments and soils potentially affected by wastes deposited in the impoundments. With unmixing methods, source types are characterized as end members, and subsequent calculations attribute proportionality of each source type within each sample. The application of unmixing to environmental data sets builds on a history of its use across a broad range of disciplines including radio signal analysis, food product evaluation and development of fuel blends. Application to source evaluation in contaminated environments has been growing nationally for the last decade, and examples are available in the published literature, and on line.

REFERENCES[6]

Barabas, N., P. Goovaerts, and P. Adriaens, 2004. Modified Polytopic Vector Analysis To Identify and quantify a Dioxin Dechlorination Signature in Sediments. 2. Application to the Passaic River. *Environ. Sci. Technol.* 38:1821–1827.

Bright, D.A. 2001. Reducing Contaminant Sources to Municipal Wastewater Treatment Biosolids: Dioxins/Furans and Mercury. Prepared for Environment Canada; BC Ministry of Environment, Lands and Parks; Capital Regional District; Greater Vancouver Regional District; Comox-Strathcona Regional District; Regional District of Nanaimo; Village of Cumberland; Squamish-Lillooet Regional District; City of Kelowna. EC/GB-00-MMI.

[6] Other than those with Web addresses provided.

Du, S., T.J. Belton, and L.A. Rodenmburg, 2008. Source Apportionment of Polychlorinated Biphenyls in the Tidal Delaware River. *Environ. Sci. Technol.* 42: 4044–4051.

Du, S., and L.A. Rodenmburg, 2007. Source Identification of Atmospheric PCBs in Philadelphia/Camden Using Positive Matrix Factorization Followed by the Potential Source Contribution Function. *Atmospheric Environment* 41: 8596–8608.

Ehrlich, R., R.J. Wenning, G.W. Johnson, S.H. Su, and D.J. Paustenbach, 1994. A Mixing Model for Polychlorinated Dibenzo-*p*-Dioxins and Dibenzofurans in Surface Sediments from Newark Bay, New Jersey Using Polytopic Vector Analysis. *Arch. Environ. Contam. Toxicol.* 27: 486–500.

Grundy, S.L., D.A. Bright, W.T. Dushenko, M. Dodd, S. Englander, K. Johnston, D. Pier, and K.J. Reimer, 1997. Dioxin and Furan Signatures in Northern Canadian Soils: Correlation to Source Signatures Using Multivariate Unmixing Techniques. *Chemosphere* 34: 1203–1219.

Imamoglu, I., K. Li, E.R. Christensen, and J.K. McMullin, 2004. Sources and Dechlorination of Polychlorinated Biphenyl Congeners in the Sediments of Fox River, Wisconsin. 38(9):2574–2583.

Mas, S., A. de Juan, R. Tauler, A.C. Olivieri, and G.M. Escandar, 2010. Application of Chemometric Methods to Environmental Analysis of Organic Pollutants: A Review. *Talanta* 80:1052–1067.

Mostert, M.M.R., G.A. Ayoko, and S. Kokot, 2010. Application of Chemometrics to Analysis of Soil Pollutants. *Trends in Analytical Chemistry* 29(5):430–445.

Rachdawong, P., and E.R. Christensen, 1997. Determination of PCB Sources by a Principal Component Method with Nonnegative Constraints. *Environ. Sci. Tech.* 31(9):2686–2691.

Stout, S.A. and T.P. Graan, 2010. Quantitative Source Apportionment of PAHs in Sediments of Little Menomonee River, Wisconsin: Weathered Creosote versus Urban Background. *Environ. Sci. Tech.* 44(8):2932–2939.

Tian, F., J. Chen, X. Qiao, X. Cai, P. Yang, Z. Wang, et al., 2008. Source Identification of PCDD/Fs and PCBs in Pine (*Cedrus deodara*) Needles: A Case Study in Dalian, China. *Atmos. Environ.* 42:4769–4777.

Wait, A.D. 2001. Environmental Forensic Chemistry and Sound Science in the Courtroom. *Fordham Environmental Law Journal* 12:293–328.

APPENDIX D
EPA COMMENTS ON SJRWP SAMPLING AND ANALYSIS PLAN: SOIL STUDY AND RESPONSES

EPA Comments on SJRWP Sampling and Analysis Plan: Soil Study and Responses[a]

Comment No.	Section	Page	Line	Comment	Response to Comment - Proposed Revision
Revised Draft SAP: Soil Study:					
1	Whole Document			EPA considers surface soil as the top 6 inches of soil under certain conditions, such as non-residential site uses (e.g., see highlighted text from EPA, 2001). In addition, typical activities for commercial/industrial site use may result in direct exposure to soils at depths of up to two feet (e.g., see highlighted text from EPA, 2000). TCEQ typically requires that surface soil exposure to ecological receptors is modeled by evaluating the top 6 inches of soil (e.g., see Issue 18 of TCEQ, 2005). Thus, all soil samples will be identified as and collected from: • surface soil: 0 to 6 inches • shallow subsurface soil: 6 to 12 inches • deep subsurface soil: 12 to 24 inches EPA. 2000. Data Quality Objectives Process for Hazardous Waste Site Investigations. EPA QA/G4HW, Jan 2000, EPA/600/R-00/007. EPA. 2001. Supplemental Region 6 Risk Assessment Guidance. Region 6 Risk Assessment Peer Review Committee. August. TCEQ. 2005. Position Paper on Common Issues Encountered During the Review of Ecological Risk Assessments. Remediation Division. September: http://www.tceq.state.tx.us/assets/public/remediation/positionpaper.pdf	The depth increments indicated by the Soil SAP, and the terminology used to refer to them, will be modified according to the request. The appropriate guidance will be cited in the discussion of the rationale.
2	1.4.1 Site Description		Last sentence of the second paragraph	Reword the last sentence of the second paragraph to: 'Related uncertainties will be addressed in the Soil SAP Addendum to be submitted on behalf of IPC.' and delete the last paragraph. The contents of the last paragraph are unnecessary for implementation of the work plan at this time.	The revision will be made as requested.
3	1.4.1.4 Surrounding Land Uses	Page 9		The text at the bottom of page 9 states that a sandy intertidal zone is present along the shoreline throughout much of the Site (Figure 2). This is not evident in the figure indicated. We suggest that Figure 2 and/or Figure 4 be modified to indicate the normal high tide line (or something similar) to better distinguish between that which is normally sediment versus that which is normally soil. This will also facilitate a more informed evaluation of the adequacy of the number and location of the proposed soil sampling locations. Additionally, a figure indicating the scope/border of the TCRA effort at Area 5 should also facilitate a more informed evaluation of the proposed soil sample locations.	Figure 4 will be modified as requested.
4	1.4.2.2 Background Soil Data			Table 1 displays the TEQ_{DF} for soil samples collected in the Houston area by the TMDL program. Please verify the TEQ calculations. For instance we were not able to duplicate the TEQ calculated for transitional soil (SS-8, 5/30/2005; and SS-16; 3/15/2005). Additionally we were not able to locate the data indicated as "soil" in the report referenced.	Use of the hardcopy TMDL report as a source of information on environmental samples may generate results that are different from those in the Site database. Reviewers may recall that inconsistencies between hardcopy reports and electronic data were discovered early in 2010. Following careful review of these discrepancies, it was decided that the database was the more accurate source. As a result of this discussion, Section 3.3. of the RI/FS Work Plan establishes that "the Site geodatabase will serve as the source compendium for all environmental data." The values for the Table 1 were derived from the Site geodatabase, and are therefore considered to be

Comment No.	Section	Page	Line	Comment	Response to Comment - Proposed Revision
					correct. Also, the database calculates TEQs automatically, so if there is an error in the code, it would be apparent in all the TEQ values. It may be that the source used by the reviewer Text will be revised to clarify the differences between the Table and the TMDL program reports.
5	1.5 Chemicals of Potential Concern and Soil Analytes			Regarding secondary COPCs, we suggest this discussion also acknowledge that a COPC Technical Memorandum is forthcoming (response to USEPA comments on the Tissue SAP) for the northern impoundments.	The COPC technical memorandum will be referenced as requested.
6	1.6.1 Nature and Extent			Based on topography, nearby sand processing activities, and wind directions, site related contaminants could have been released to the vegetated upland area located immediately west of Area 2. Please propose steps or a plan to collect samples in this area.	In light of the recent data for the Big Star soils which show relatively low levels of soil contamination up to 1 ft deep, it is considered unlikely that wind transport could have generated significant soil contamination in the area west of the Big Star property. Additional samples in that area are not needed.
7	1.6.1 Nature and Extent			In addition, please propose steps or a plan to collect additional samples in Area 4 and to the west of Area 4, to reflect the same sample density as Area 1, 2, 3, 5.	Available data indicate that concentrations of primary and secondary COPCs in soils in Area 4 (the TxDOT right of way) are very low, below conservative screening levels and generally within the range of background. Additional samples in Area 4, or to the west of Area 4 are not needed.
8	1.8.1.2 Sample Collection Design (Background Conditions) and Figure 5			The proposed background sampling locations of River Terrace Park, Lynchburg Ferry Site, and the San Jacinto Battleground Park are not appropriate as these locations are either dredge fill islands or sites likely impacted by the river floods and/or fires. However, we do agree with Respondents on the proposals of I-10 at Beltway 8 and Burnett Park, and further recommend Banana Bend as a third location. In addition, Respondents should provide research information on fill used or any routine application of soil additives (i.e., fertilizers, compost, sewage sludge, etc.) at these three locations (i.e., I-10 at Beltway 8, Burnett Park, and Banana Bend).	An important criterion for background areas is that the area is directly adjacent to a freeway. The two areas proposed in the draft Soil SAP, and acceptable to EPA, will be used for background samples. Additional research will be conducted to identify sources of information on soil management in Beltway 8 and Burnett Park. Any information that is obtained will be provided in the revised Soil SAP.
9	1.8.1.2 Sample Collection Design (Background Conditions) and Figure 5			The document suggests that the ten' new' background samples will be supplemented with the ten existing soil samples in residential and urban areas that were collected in 2004-2005 for the TMDL program (as discussed in Section 1.4.2.2). We do not support using the TMDL soil data as a supplement to the soil background data set because questions such as soil depth, specific sample locations, and laboratory quality assurance information remain unresolved. Instead, we recommend collection of ten background samples (at 0-6" and 6"-12" intervals) at each of the background locations identified in the previous comment (i.e., I-10 at Beltway 8, Burnett Park, and Banana Bend).	It is correct that the TMDL data for background soils are not ideal for use in this project because of differences in the methods used to collect those samples. However, other uncertainties cited by the reviewer can be addressed: • Locations: the map of sample areas provided by the TMDL program can be provided in the revised Soil SAP • QA/QC: Respondents are currently pursuing laboratory data packages from D. Rifai. We have successfully done this for other data. Questions about sampling methods will also be presented to her. In addition, results presented in Table 1 are consistent with results of an urban background study conducted recently in Denver, which shows a distribution of TEQDF very similar to that of the data in Table 1 (USEPA 2001) . For these reasons, Respondents consider the available data, when combined with the proposed new background samples, to provide an appropriate representation of background for the purposes described in the SAP. USEPA. 2001. Denver Front Range Study. Dioxins in Surface Soil. Study 1: Characterization of Dioxins, Furans and PCBs In Soil Samples Collected from the Denver Front Range Area. Report prepared by USEPA Region 8 with technical

Comment No.	Section	Page	Line	Comment	Response to Comment - Proposed Revision
					assistance from Syracuse Research Corporation, Inc. and Gannett Fleming, Inc. July, 2001.
10	1.8.1.3 Analytic Approach	Page 27		Regarding the second full bulleted paragraph on page 27, the discussion summarizes a plan to use a pattern-matching approach to evaluate sediments or wastes from within the impoundments, and both Site and background soil samples, to identify any pattern characteristic (using data for dioxins and furans) of the impoundment. These results, according to the discussion, will be used to determine the contribution of sediments with this pattern to soils in the upland area west of the impoundments, and in the vicinity of I-10. The Respondents should provide more details regarding the proposed pattern-matching approach (reference, example calculations, etc.) and the 'end member'.	Additional description of the approach to dioxin fingerprinting and pattern matching will be provided in the discussion of the analysis approach, in the DQO section.
11	1.8.1.3 Analytic Approach			Section needs to be revised to reflect changes in background sample locations and that EPA and TCEQ prefer the use of the Upper Prediction Limit for the determination of a background statistic. Also, the EPA reference indicated (USEPA 2005a) does not appear to be the correct reference for a discussion of background determinations and statistical comparisons for soil data.	EPA Guidance does not state a preference for the upper prediction limit (UPL); the citation for the guidance document will be corrected. However, both the RI/FS Work Plan and the Sediment SAP acknowledge the TCEQ preference for the UPL, and it will be acknowledged in this SAP as well. The Soil SAP will be revised to indicate that evaluation of concentrations of chemicals on the Site relative to background will include consideration of the UPL.
12	1.8.1.3 Analytic Approach	Page 26		The discussion in the second full bulleted paragraph on page 26 and continuing on to the top of page 27 reflects that analysis of some of the samples from Area 2 will be 'conditioned' upon an exceedence of the EPA's interim PRG for residential soils in the majority of both surface intervals at the sample locations that are initially analyzed. How does this threshold compare with that anticipated for a terrestrial ecological receptor (i.e., if a particular exposure pathway for a terrestrial receptor/guild is more sensitive than the interim human health PRG, what would be the next steps)?	The revised sampling design does not include the conditional samples, so the evaluation referenced in the comment will not be needed.
13	1.5 Chemicals of Potential Concern and Soil Analytes			Previous comments submitted still apply here. From the Sediment SAP and Tissue SAP Comments, whether or not a secondary COPC will not be evaluated in the BLRA will depend on the relative concentrations between the secondary COPC and dioxins and furans for each sample. As stated in this section, "…is likely to be addressed…" does not prove that the secondary COPCs that correlate with dioxins/furans are/have been addressed. Secondary COPCs that are detected and fail the risk-based screens will need to be included in risk calculations to prove that they are not an issue.	The following language currently appears in the Soil SAP, and is similar to related language that has been used in other SAPs to address this concern: "As noted for sediment and tissue COPCs, these decision rules apply unless additional information indicates that a COPC may or may not be present at elevated levels in soil on Site as a result of activities associated with sediments that may have been affected by waste from the impoundments that are north of I-10." Regarding the final statement in this comment: Respondents have previously proposed that the need for risk assessment for each chemical be addressed by the COPC Technical Memorandum. This will allow and informed and collaborative decision for each chemical that takes into account more than a pass/fail screening assessment. The COPC Technical Memorandum, and its intended uses on this project, will be noted in the text of the revised Soil SAP.
14	1.6.2 Human and Ecological Exposures			The trespasser has been left out, suggesting that they do not have a complete and significant soil exposure pathway. What is the justification for this?	The trespasser will be added to the text under Section 1.6.2 to clarify that there is a potentially complete exposure pathway. This is consistent with the CSM for human health presented in the RI/FS Work Plan.

EPA Comments on SJRWP Sampling and Analysis Plan: Soil Study and Responses[a]

Comment No.	Section	Page	Line	Comment	Response to Comment - Proposed Revision
15	1.8.1.1.2 Exposure Assessment			Direct contact has been defined here as ingestion and dermal contact. What about inhalation?	In the CSM for human health presented in the RI/FS Work Plan inhalation exposure is associated to airborne particulates, which includes suspended soil particles. Regardless of nomenclature, these exposure routes are considered additive in a human exposure assessment.
16	Table 7			Note (a) denotes that the HHRA ACGs are the USEPA Region 3 Soil PRGs. Please note that there are numerous NA's listed in the table. Applicable TRRP values should also be considered, as there are values present for the compounds which have an NA.	The table will be revised as requested.
17	Figure 3			There are several human receptor pathways that are considered complete, but minor. If a pathway is considered to be complete, then it needs to be evaluated.	Comment noted.

a - A draft Sampling and Analysis Plan: Soil Study (Soil SAP) was submitted to EPA on September 10, 2010. On October 22, 2010, EPA sent a letter to the project coordinator directing Respondents to revise the Draft Soil SAP to address the area south of I-10, and to resubmit the revised Soil SAP on November 8, 2010. These technical comments refer to the November 8, 2010 submittal.